Caparica • June 22—25, 2008 • PORTUGAL

7th IBERIAN vacuum MEETING

& 5th European Topical Conference on Hard Coatings

Chairmen

Orlando M.N.D Teodoro
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José L. de Segovia
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Abstract Book

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FACULDADE DE CIÊNCIAS E TECNOLOGIA
UNIVERSIDADE NOVA DE LISBOA

Book of Abstracts

7th Iberian Vacuum Meeting
&
5th European Topical Conference on Hard Coatings

Welcome

Advances in vacuum science and technique are the major reason for the remarkable achievements reached in the last decades, in many scientific fields. The ability to produce high vacuum, allowed the study of critical reactions in gas phase; generation of ultra-high vacuum, lead to the foundation of nanotechnology and to the establishment of surface science; and creation of extreme high vacuum is improving the performance of large accelerators.

Although, further progresses in vacuum science are still expected, its applications still have much to develop. Every day, new scientific and technical accomplishments are reported in fields as nanocatalysis, functional thin films or plasma applications.

Therefore, scientists and engineers, whose research is vacuum dependent, have much to share and to discuss in a forum as the Iberian Vacuum Meeting intends to be. Special attention will be given in a parallel conference to the application on hard coatings, due to its increasing relevance in the production of new devices and tools.

Welcome all of you who continue using vacuum, no matter the application. Welcome to the 7th Iberian Vacuum Meeting and 5th European Topical Conference on Hard Coatings at Caparica, June 22-25, 2008. Your contribution is most welcome and your presence surely rewarded.

Orlando M.N.D Teodoro

José L. de Segovia

Organization

The 7th Iberian Vacuum Meeting & 5th European Topical Conference on Hard Coatings is a joint organization of the Portuguese and Spanish Vacuum Societies. The meetings are held typically every 2-3 years alternatively in Portugal and in Spain.

The first meeting was held in Braga in 1986. Later, in 1994 a new conference series on hard coatings join the Iberian meeting.

Conference Topics are:

- Vacuum Science & Technique
- Applied Surface Science;
- Surface Engineering;
- Thin Films;
- Electronic Materials & Processing;
- Nanometer Structures;
- Surface Science;
- Plasma Science and Technique;
- Hard Coatings.

Committees:

Local Organizing Committee:

- Orlando M.N.D Teodoro (DF FCT/UNL)
- Ana Rita Canário (DF FCT/UNL)
- Cristina Groba (CIIDI, FCT/UNL)
- Helena Godinho (DCM FCT/UNL)
- Gregoire Bonfait (DF FCT/UNL)
- Isabel Catarino (DF FCT/UNL)
- Isabel Ferreira (DCM FCT/UNL)
- Isabel Fonseca (DQ FCT/UNL)
- José Luís Ferreira (DF FCT/UNL)
- Reinhard Schwarz (DF IST(UTL)
- Yuri Nunes (DF FCT/UNL)

Scientific Committees:

7th Iberian Vacuum Meeting

- Ana Rego (IST, Lisboa)
- Asunción Fernández (ICM, Sevilla)
- Augusto M.C. Moutinho (UNL, Lisboa)
- Carlos Sá (FEUP, Porto)
- Elisa Roman (ICM, Madrid)
- Florinda Costa (UA, Aveiro)
- Gregoire Bonfait (UNL, Lisboa)
- Isabel Montero (ICM, Madrid)
- Jose A. Martin-Gago (ICM, Madrid)
- Orlando Teodoro (UNL, Lisboa)
- Reinhard Schwarz (IST, Lisboa)
- Rodrigo Martins (UNL, Lisboa)
- Vasco Teixeira (UM, Guimarães)

5th European Topical Conference on Hard Coatings

- Albano Cavaleiro (FCTUC, Coimbra)
- Alberto Alberdi (Tekniker, Gipuzkoa)
- Arturo Lousa (FFUB, Barcelona)
- Jose Antonio Garcia (AIN, Navarra)
- Jose M. Albella" (ICM, Madrid)
- Juan Carlos Sánchez López (ICM, Sevilla)
- Manuel J.P. Maneira (UNL, Lisboa)
- Pedro Alpuim (UM, Guimarães)
- Rui Silva (UA, Aveiro)

Secretariat:

Engª Ana Fonseca

Phone: (+351) 212 948 576, Fax : (+351) 212 947 879, Email: metrovac@fct.unl.pt

Venue

Venue is at the Caparica Campus of Universidade Nova de Lisboa on June 22-25, 2008. This is very close to the beaches of Costa da Caparica only 20 minutes far from Lisbon. The weather in this season is usually perfect to enjoy the beauty of South Lisbon seaside landscape. Companions are welcome, since they will also have much to enjoy.

A high level scientific program has been prepared with 23 reputed scientists invited to give talks on the state-of-art of science and technology of the conference topics.

Students, both graduation and pos-graduation are highly encouraged to be present. Submitted papers will be published in Vacuum after peer review.

About Almada and Caparica

The City

Sharing the magnificent estuary of the Tagus with Lisbon, the city of Almada offers, those who visit it, the tranquillity of the narrow streets of Old Almada and its belvederes, large windows over the river and the capital.

Forming part of the city, Pragal, grew on the hillside, topped by the monument to Christ the King, whilst to the south, Feijó has green areas linking the fields to the urban fabric.

However, for those who want to know more about the Municipality of Almada there is nothing like paying a visit to the City Museum, housed at the old Quinta dos Frades, in Cova da Piedade.

To live in the city is to feel it throbbing, to feel the art of good hosting of its people and the joy of youth. It is to stroll its streets and squares finding public art, resting in its parks and gardens, enjoying the coolness of an esplanade.

The Riverfront

In the picturesque inlets of the riverfront, one finds the places that keep the memories of river fishing in the gastronomy offered.

Until the middle of the last century Cacilhas was a summer location for the people of Lisbon who when they arrived opted for the river beaches or for the adventure of the donkey rides. Today, the old Ginjal Quay invites you for a peaceful stroll up to the Jardim do Rio (River Garden) that frames Fonte da Pipa (Fountain of the Cask), leads to the Naval Nucleus and challenges you to a trip on the panoramic lift.

In Trafaria, those who arrive by boat are welcomed by an impressive, but friendly fort. On land surrender yourself to poetry along the riverfront path and discover the beauty of the other margin from among the colours of the small fishing boats, through the tangle of ropes and the transparency of the nets.

The Atlantic Front

The quality of the landscape together with the vast stretches of sand where fishing relieves the visitor's tedium meant the Atlantic Front was, from early on, a place destined for leisure and to spend summer. Even with the Costa da Caparica improvement works that are currently taking place, do not miss the festival of colours put on by the "Meia-Lua", back from fishing at sea and the boards and sails with which the nautical sports enrich the palette of the horizon.

At the top of the cliff, the Convento dos Capuchos (Convent), with its garden and belvedere invites you to watch the spectacle of the sunset over the waters of the Atlantic.

Registration

	Before May 16	**After May 16**	**1 day conference**
Senior:	320€	450€	200€
Student:	180€	250€	100€
Companion:	70€	80€	
Fee includes the Welcome reception on Sunday, 3 lunches, coffee-breaks, excursion, 1 special dinner and full access to both conferences (for companions: welcome reception, conference trip and 1 special dinner)			Fee includes 1-day full access to both conferences and 1 lunch and coffee breaks.

Fees will allow attendance to both conferences.

Payments should made to

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Cancellation of Conference Participation:

- All cancellations should be made by fax or by e-mail.
- Your payment less 50 Euros will be reimbursed, if a cancellation is received June 1, 2008.
- 50% of the registration fee payment will be reimbursed, if a cancellation is received after this date and before June 15.
- No reimbursement will be made after June 15, 2008.

Insurance:

The Organization Committee will not be responsible for any medical expenses, lost property, or accidents during the meeting and we strongly recommend that participants make all necessary insurance arrangements for themselves.

Lunches

Lunches will be provided in a special line at the *Cantina* starting at 13h10, from Monday to Wednesday.

Tickets for every lunch are included in the conference bag. Please bring the tickets with you on leaving to lunch.

Parking

Parking inside the campus will be allowed to all the attendees. Please show your badge at the entrance when entering by car. Please, do not use the reserved places in front of the conference building.

Accommodation

All enquiries relating accommodation should be directly addressed to:

Viagens Abreu - Congress Department, Att. Mrs. *Maria de Jesus*
Av. 25 de Abril 2 - Edifício Abreu
2799-556 Linda-a-Velha - Portugal
E-Mail: mbacalhau.lisboa@abreu.pt, Tel: +351 21 415 61 24, Fax: + 351 21 415 63 83

Social Program

Sunday 22, 18h00 to 20h00—	Welcome reception
Tuesday 24, 16h20 to 20h00—	Conference Excursion and Wine Tasting at *Caves José Maria da Fonseca*
Tuesday 24, 20h00 to 23h00—	Conference Dinner

Please bring the printed invitation included in the conference bag to the conference excursion and dinner.

If you have no ticket for the conference dinner you may still buy one at the secretariat.

Contributed papers

Posters

An area of approximately 1m wide is reserved for each poster. Therefore, the standard A0 vertical (≈1.20 m height x 0.85m width) is the recommended size.

Although the poster session will be only on Tuesday 14h20m to 16h00m, it is recommended that posters should be displayed from Monday to Wednesday morning. It is expected that, at least, one author should be present near the poster during the poster session. Pins and tape to hold the posters will be available in the secretariat.

Proceedings

All authors are encouraged to submit a manuscript for publication. Contributions are welcomed from both academic and industrial sectors. They must present original and previously unpublished advances on knowledge and /or technology.

Selected papers will be published in a special volume (consecutively numbered) of Vacuum after peer review. The format and layout will be the same as for regular issues of the Journal.

The size of the articles will be 6 printed pages each for invited papers and 4 pages each for contributed papers, respectively. Contributions in the form of extended abstracts cannot be accepted. Publication date will be no later then August 2009. Guide for authors is available for download at the conference website.

Manuscripts (3 copies) should be delivered during the meeting in the conference secretariat.

Sponsors

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Reitoria da Universidade Nova de Lisboa

French Embassy in Portugal

Exhibition

Manufacturers and representatives of scientific equipment will exhibit their products. This will give to the attendees the opportunity to obtain information about the latest developments in their products.

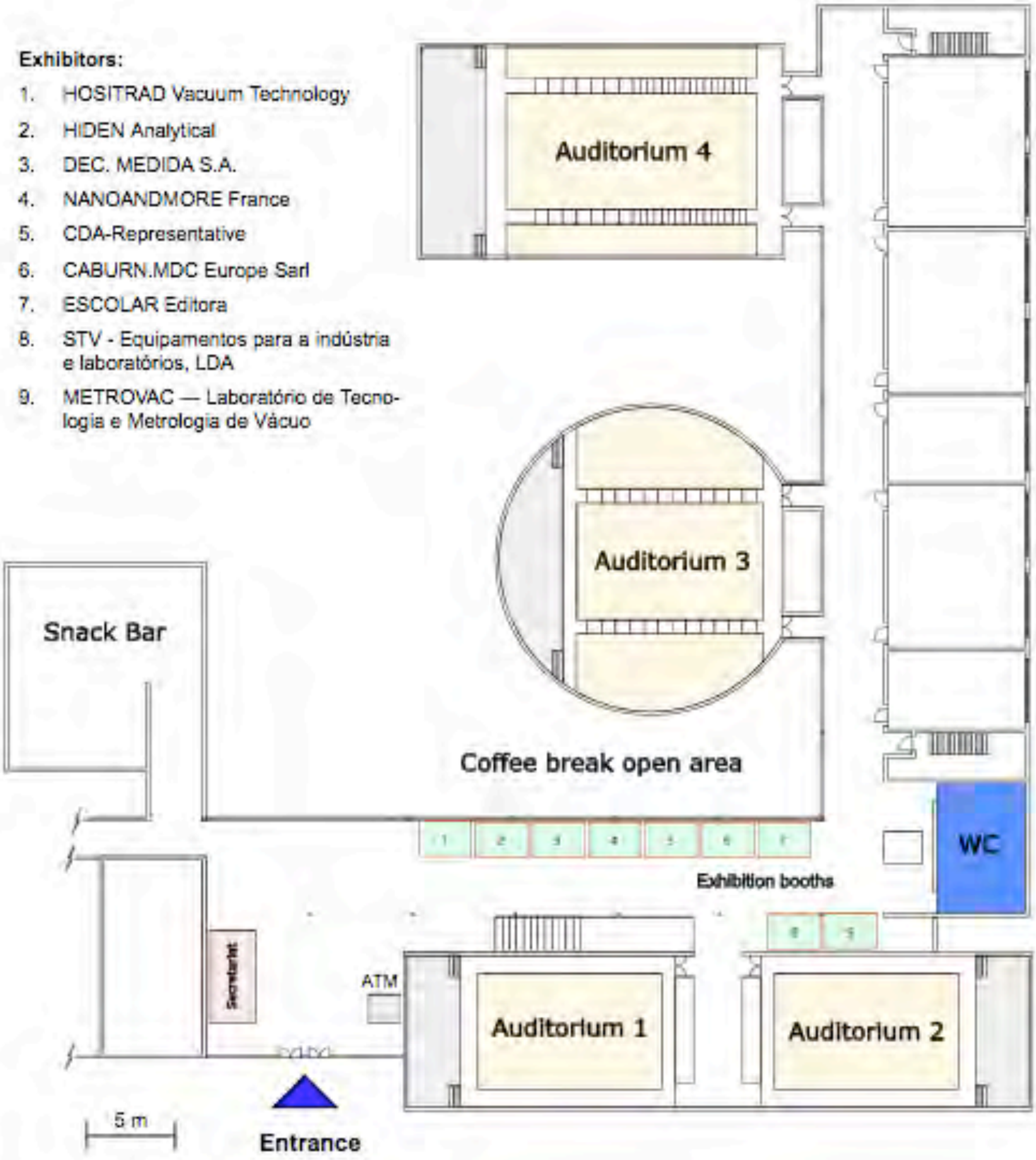
1. Hositrad Vacuum Technology
   info@hositrad.nl
2. Hiden Analytical
   info@hiden.co.uk
3. Dec. Medida S.A.
   geral@decmed.pt
4. Nano and More France
   sales@nanoandmore.fr
5. CDA-Representative
   deazevedo_carlos@yahoo.fr
6. Caburn - MDC Europe Sarl
   asaubert@caburn.fr
7. Escolar Editora
   vhugo@escolareditora.com

8. STV - Equipamentos para a industria e laboratórios,LDA

   geral@stv.pt

9. Metrovac- Laboratório de Tecnologia e Metrologia de Vácuo

   metrovac@fct.unl.pt



Invited Speakers

Plenary Speakers (confirmed):

1. J.L. de Segovia, Instituto de Ciencia de Materiales de Madrid (former IUVSTA President), Spain
2. Guglielmo Fortunato, Flexidis and IMM- CNR, Italy
3. Ron Naaman , The Weizmann Institute of Science, Israel
4. Geoff Thornton, London Centre for Nanotechnology, University College London, UK
5. Marcel Rost, Kamerlingh Onnes Laboratory, Leiden University, The Netherlands
6. Akhlesh Lakhtakia, Pennsylvania State University, University Park, USA

Invited Speakers (7th IVM)

7. Pierre Jamotton, Facility Department, Low Temperatures Laboratory, Centre Spatial de Liege, Université de Liège, Belgium
8. Wolfgang Jitschin, University of Applied Sciences Giessen-Friedberg, Germany
9. Lluis Miralles, CELLS Engineering Division, Barcelona, Spain
10. João Pedro Conde , Instituto Superior Técnico, and INESC Microssistemas e Nanotecnologias, Lisbon, Portugal
11. Jose A. Martín Gago, Instituto de Ciencia de Materiales de Madrid, Spain
12. Nuno Peres, Department of Physics, Universidade do Minho, Portugal
13. Jatin K. Rath, SID-Physics of Devices, RvdG Laboratory, Utrecht University, The Netherlands
14. Olinda Conde, Department of Physics, Universidade de Lisboa, Portugal

Invited Speakers (5th ETCHC)

15. Ramón Escobar Galindo, Centro de Microanálisis de Materiales, Universidad Autónoma de Madrid, Spain
16. Luis Rebouta, Department of Physics, Universidade do Minho, Portugal
17. Tomas Polcar, University of Coimbra, Departamento de Engenharia Mecânica, Portugal
18. Johan Böhlmark, CEO Chemfilt Ionsputtering AB Linköping, Sweden
19. Wolfgang Diehl, Fraunhofer-Institut für Schicht- und Oberflächentechnik, Braunschweig, Germany
20. Fabien Bénédic, Laboratoire d'Ingénierie des Matériaux et des Hautes Pressions, CNRS, Université Paris 13, France
21. Philippe Steyer, Laboratoire de Physico-Chimie Industrielle, INSA de Lyon, France
22. Rui Silva, CICECO, Ceramics & Glass Eng. Dept. University of Aveiro, Portugal
23. Rafael Rodriguez, AIN-Centro de Ingeniería Avanzada de Superficies, 31191 Cordovilla, Pamplona, Spain

Program at a glance:

7th Iberian Vacuum Meeting

June	Sunday 22	Monday 23		Tuesday 24	Wednesday 25
8h00m	***Arrival***	Registration open			
9h00m		Opening ceremony (10 min) Plenary session 1 “History of Vacuum” by J.L. de Segovia		Plenary session 4 “Nanoscale science on metal oxide surfaces” by Geoff Thornton	Plenary session 5 “In-situ, Real-time Observation of Thin Film Deposition: Roughening, Zeno, Grain Boundary Crossing Barrier, and Steering” by Marcel Rost
10h00m		Plenary session 2 “Low temperature polysilicon TFTs on polyimide substrates for flexible electronics” by G. Fortunato		Invited 04	Invited 07
10h20m					
10h40m				Oral 12	Oral 14
11h00m		Coffee break		Coffee break	Coffee break
11h20m		Oral 01	Oral 06	Invited 05	Invited 08
11h40m		Oral 02	Oral 07		
12h00m		Oral 03	Oral 08	Invited 06	Plenary session 6 “Elastodynamics of Inorganic and Polymeric Sculptured Thin Films” by Akhlesh Lakhtakia
12h20m		Oral 04	Oral 09		
12h40m		Oral 05	Oral 10	Oral 13	
13h00m		**Lunch**		**Lunch**	**Lunch**
14h20m		Plenary session 3 “Cooperative Molecular Field Effect- A Source for New Electronic Properties at Interfaces” by Ron Naaman		Poster session	Conference ends *Departure*
15h20m		Invited 01			
15h40m					
16h00m		Coffee break		Coffee break	
16h20m		Invited 02		*Excursion*	
16h40m					
17h00m		Oral 11			
17h20m		Invited 03			
17h40m					
18h00m	*Welcome reception* (Registration open)				
20h00m				*Conference dinner*	

June	Sunday 22	Monday 23	Tuesday 24	Wednesday 25
8h00m	***Arrival***	Registration open		
9h00m		Opening ceremony (10 min) Plenary session 1 “History of Vacuum” by J.L. de Segovia	Plenary session 4 “Nanoscale science on metal oxide surfaces” by Geoff Thornton	Plenary session 5 “In-situ, Real-time Observation of Thin Film Deposition: Roughening, Zeno, Grain Boundary Crossing Barrier, and Steering” by Marcel Rost
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10h20m				
10h40m			Oral 03	Oral 05
11h00m		Coffee break	Coffee break	Coffee break
11h20m		Invited 01	Invited 07	Oral 06
11h40m				Oral 07
12h00m		Invited 02	Invited 08	Plenary session 6 “Elastodynamics of Inorganic and Polymeric Sculptured Thin Films” by Akhlesh Lakhtakia
12h20m				
12h40m		Oral 01	Oral 04	
13h00m		**Lunch**	**Lunch**	**Lunch**
14h20m		Plenary session 3 “Cooperative Molecular Field Effect- A Source for New Electronic Properties at Interfaces” by Ron Naaman	Poster session	Conference ends *Departure*
15h20m		Invited 03		
15h40m				
16h00m		Coffee break	Coffee break	
16h20m		Invited 04	*Excursion*	
16h40m				
17h00m		Oral 02		
17h20m		Invited 05		
17h40m				
18h00m	*Welcome reception (Registration open)*			
20h00m			*Conference dinner*	

Program

Monday Morning

	7th IVM		5th ETCHC
9h00m	**Room: Auditorium 3, Chair: Orlando Teodoro**		
	Opening Cerimony		
9h10m	**Plenary session 1** J.L. de Segovia, Instituto de Ciencia de Materiales de Madrid (former IUVSTA President) “History of Vacuum”		
10h00m	**Plenary session 2** G. Fortunato, Flexidis and IMM- CNR, Italy “Low temperature polysilicon TFTs on polyimide substrates for flexible electronics"		
11h00m	**Coffee break**		
11h20m	**Chair: R. Schwarz and Elisa Roman** **Room: Auditorium 1**	**Chair: Florinda Costa and J. Martin-Gago** **Room: Auditorium 2**	**Chair: Arturo Lousa and Rui Silva** **Room: Auditorium 3**
	7IVM Oral 01 C. Petitjean, D. Horwat and J.F. Pierson “Structure and properties of sputtered Ag-Cu-O films: effect of the oxygen flow rate and of the target chemical composition”	**7IVM Oral 06** Baogui Shi, John L.Sullivan and Sayah O.Saied “Sputtered Carbon coating Characterization and Bias voltage effect”	**5ETCHC Invited 01** Rámon Escobar Galindo , R. Gago, A. Lousa, C .Palacio, J.M. Albella, Autonoma University of Madrid, Spain “Advanced characterization of nanometer multilayers: depth profiling comparison of RBS, SIMS, XPS and GDOES”
11h40m	**7IVM Oral 02** C.A.A. Ghumman, A.M.C. Moutinho, V. Vassilenko and O.M.N.D. Teodoro “Investigation on renal stone analysis with ToF-SIMS”	**7IVM Oral 07** John L.Sullivan “Physical properties of ultra fast deposited micro and nano thickness amorphous hydrogenated carbon films for medical devices and prostheses”	
12h00m	**7IVM Oral 03** Hugo Águas, Sergej A. Filonovich, Iwona A. Bernacka, Elvira Fortunato and Rodrigo Martins “Highly conductive p-type nanocrystalline silicon films deposited by rf-PECVD using silane and trimethylboron mixtures at high pressure”	**7IVM Oral 08** A. Cavaleiro Adhesion problems related with deficient ion cleaning of the substrates	**5ETCHC Invited 02** Luis Rebouta, F. Vaz(a), S. Carvalho and K. Shein, Department of Physics, Universidade do Minho, Portugal “Superhard nanostructured coatings: structure and properties relations”
12h20m	**7IVM Oral 04** S. Carmona, C. Palacio, O. Sánchez, M. Aguilar, C. Falcony, J. Guzmán, M. Canseco, G. Alarcón and M. García “Interface properties of Al_2O_3 thin films deposited on c-Si by pulsed ultrasonic sprayed pyrolysis”	**7IVM Oral 09** Manuel Evaristo, Tomas Polcar and Albano Cavaleiro “Synthesis and structural properties of W–Se–C coatings deposited by PVD in reactive and non-reactive processes”	
12h40m	**7IVM Oral 05** António Vieira, Maria Machado Angelo Yializis and Gordon Goodyear “High speed surface functionalization of woven and non woven textiles”	**7IVM Oral 10** A.L. Shaula, V.A. Kolotygin, J.C. Oliveira, V.V. Kharton and A.A. Cavaleiro “Protective YSZ-based thin films deposited by RF magnetron sputtering”	**5ETCHC Oral 01** Alberto Alberdi, Beatriz Díaz. Marcos Marín, Josu Goikoetxea, Unai Ruiz de Gopegui and Borja Coto “Synthesis and Properties of Silicon-Aluminium Based Nanocomposite Coatings”
13h00m	**Lunch**		

Monday Afternoon

	7th IVM	5th ETCHC
14h20m	**Chair: Gregoire Bonfait and Isabel Montero** **Room: Auditorium 3**	
	Plenary session 3 Ron Naaman, The Weizmann Institute of Science, Israel “Cooperative Molecular Field Effect- A Source for New Electronic Properties at Interfaces”	
15h20m	**7IVM Invited 01** Pierre Jamoton, Facility Department, Low Temperatures Laboratory, Centre Spatial de Liege, Université de Liège "Planck Cryogenic Testing in CSL Premises, Helium Partial Pressure Management"	**Chair: Arturo Lousa and Rui Silva** **Room: Auditorium 2**
		5ETCHC Invited 03 Tomas Polcar, M. Evaristo, and A. Cavaleiro, University of Coimbra, Portugal “Transition metal dichalcogenides alloyed with carbon: a new class of self-lubricating coating”
16h00m	**Coffee break**	
16h20m	**7IVM Invited 02** Wolfgang Jitschin, University of Applied Sciences Giessen-Friedberg, Germany "Precise flow measurement of abitrary gas species over a wide range"	**5ETCHC Invited 04** Johan Böhlmark, Grzegorz Greczynski, Rainer Cremer,Yutao Pei, and Ulf Helmersson, CEO Chemfilt Ionsputtering AB Linköping, Sweden "High Power Impulse Magnetron Sputtering – an overview of history, properties and current status"
17h00m	**7IVM Oral 11** I. Catarino, D. Martins, J. Afonso, L. Duband and G. Bonfait “H_2 and Ne Gas Gap Heat Switches”	**5ETCHC Oral 02** Alderson Neira, Federico Sequeda, Alexander Ruden and Juan M. Gonzáles “PVD Nitride Hard Coatings Production on Chromium Based Tool Steel Substrates”
17h20m	**7IVM Invited 03** Lluis Miralles, CELLS Engineering Division, Barcelona, Spain " ALBA Ultra High Vacuum System status”	**5ETCHC Invited 05** Wolfgang Diehl, Fraunhofer-Institut für Schicht- und Oberflächentechnik, Braunschweig, Germany "Hard coatings produced by plasma processes – technology and recent applications"

Tuesday Morning

	7th IVM	5th ETCHC
9h00m	**Chair: J.L. de Segovia and A.M.C. Moutinho** **Room: Auditorium 3**	
	Plenary session 4 Geoff Thornton, London Centre for Nanotechnology, University College London, UK "Nanoscale science on metal oxide surfaces"	
10h00m	**7IVM Invited 04** João Pedro Conde , Department of Chemical and Biological Engineering, Instituto Superior Técnico, Lisbon, Portugal "Thin-film silicon MEMS"	**Chair: Luis Rebouta and José Maria Albella** **Room: Auditorium 2**
		5ETCHC Invited 06 F. Bénédic, D. Monéger, G. Lombardi, K. Hassouni, A. Michau, M.B. Assouar, O. Elmazria, A. Gicquel, P. Alnot, Laboratoire d'Ingénierie des Matériaux et des Hautes Pressions, CNRS, Université Paris 13, France "Nanocrystalline diamond films: from growth to applications"
10h40m	**7IVM Oral 12** P. Alpuim, P.F. Rocha, S. Lanceros-Mendez, E.S. Marins, I.G. Trindade, J.G. Rocha and M.A. Carvalho "Ultra-Sensitive Shape Sensor Test Structures Based on Piezo-Resistive Doped Nanocrystalline Silicon"	**5ETCHC Oral 03** J. Romero, L.A. Yate, L. Martínez de Olcoz, J. Esteve, C. Gomez-Aleixandre and A. Lousa "Amorphous carbon/Cr_3C_2 nanocomposite thin films"
11h00m	**Coffee break**	
11h20m	**7IVM Invited 05** R. Caillard, G. Otero, L. Alvarez, C. Sánchez-Sánchez, M .F. López, J. Mendez and J.A. Martin-Gago Instituto de Ciencia de Materiales de Madrid, Spain "Structure and self-organization of organic and bio molecules on single crystal Surfaces"	**5ETCHC Invited 07** Philippe Steyer, A. Mege, G. Thollet, C. Esnouf, Laboratoire de Physico-Chimie Industrielle, INSA de Lyon, France "How to design nanostructured PVD nitrides based coatings for increasing the durability of steel?"
12h00m	**7IVM Invited 06** Nuno Peres, Department of Physics, Universidade do Minho, Portugal "The electronic properties of graphene"	**5ETCHC Invited 08** F.A. Almeida, A.J.S. Fernandes, F.J. Oliveira and R.F. Silva, Ceramics & Glass Eng. Dept. University of Aveiro, Portugal "Semi-orthogonal turning of hardmetal with CVD diamond and commercial PCD at different cutting angles"
12h40m	**7IVM Oral 13** V.Vassilenko, H.Marques, O.M.N.D.Teodoro and . A.M.C. Moutinho "Formation and thermal stability of monolayer and multilayer films by azidoacetonitile decomposition onto rhodium surfaces"	**5ETCHC Oral 04** V. Rachpech, J.F. Pierson, S. Weber and A. Billard "Effect of air annealing on the structure and the hardness of Cr-Si-N coatings deposited using a hybrid arc-magnetron process"
13h00m	**Lunch**	

Tuesday Afternoon

	7th IVM	5th ETCHC
14h20m	**Poster session**	
16h00m	**Coffee break**	
16h20m	**Excursion**	
20h00m	**Conference dinner**	

Wednesday Morning

	7th IVM	5th ETCHC
9h00m	**Chair: Alberto Alberti and Manuel Maneira** **Room: Auditorium 3**	
	Plenary session 5. Marcel Rost, Kamerlingh Onnes Laboratory, Leiden University, The Netherlands "In-situ, Real-time Observation of Thin Film Deposition: Roughening, Zeno, Grain Boundary Crossing Barrier, and Steering"	
10h00m	**Chair: Ana Rego and Juana Benavente** **Room: Auditorium 2** **7IVM Invited 07** Jatin K. Rath, SID-Physics of Devices, RvdG Laboratory, Utrecht University, The Netherlands "In-situ diagnostics in VHF PECVD process : A crucial aspect for fast fabrication of thin film silicon solar cells"	**5ETCHC Invited 09** Rafael Rodriguez, AIN-Centro de Ingeniería Avanzada de Superficies, 31191 Cordovilla, Pamplona, Spain "New trends in decorative and functional coatings"
10h40m	**7IVM Oral 14** Eduardo Alves, Nuno Barradas, F. Munnik, Teresa Monteiro, Marco Peres, Jinzhong Wang, Rodrigo Martins and Elvira Fortunato "Structural and optical properties of nitrogen doped ZnO films"	**5ETCHC Oral 05** J. A. García, Ibon Azkona, J. Fernandez de Larriona, M.J. Diaz de Cerio, R. Martínez 1, R. J. Rodríguez , R. Escobar and J.M. Albella "Oxidation behaviour of CrAlN, AlTiN and nonocomposite coatings"
11h00m	**Coffee break**	
11h20m	**7IVM Invited 08** Olinda Conde, Nicoleta Popovici and M. Luísa Paramés, Department of Physics, Universidade de Lisboa, Portugal "Pulsed Laser Deposition of Oxide-based thin films for spintronics"	**5ETCHC Oral 06** C. Louro; J.C. Oliveira, N.M.G. Parreira and A. Cavaleiro "Effect of O Addition on the Thermal Behaviour of Hard W-N Sputtered Coatings"
11h40m		**5ETCHC Oral 07** B. Skoric, D. Kakas, A. Miletic and G. Favaro "The Nano Modification of Duplex Hard Coatings with Nitrogen Ion Implantation"
12h00m	**Plenary session 6.** Akhlesh Lakhtakia, Pennsylvania State University, University Park, USA "Elastodynamics of Inorganic and Polymeric Sculptured Thin Films"	
13h00m	**Lunch & Conference ends**	

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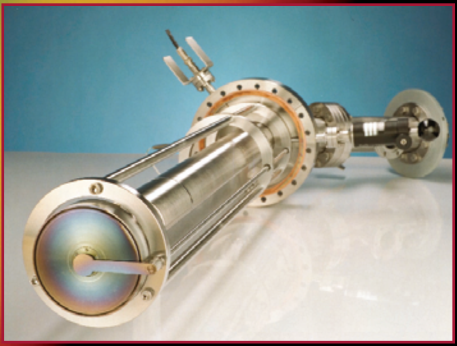

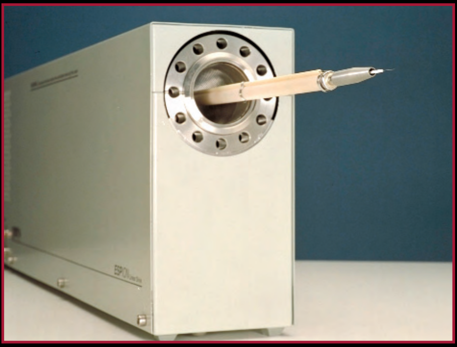

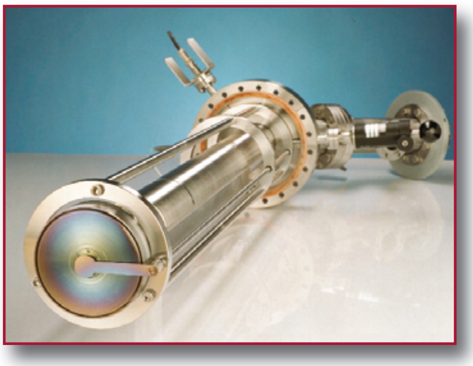

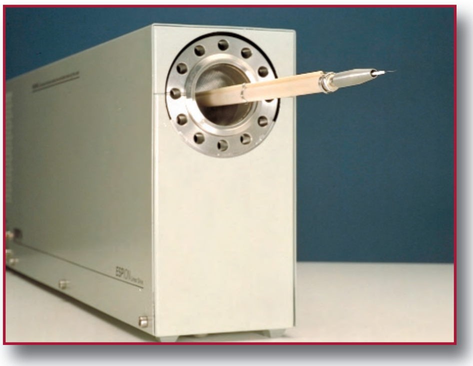

Monday Morning

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The History of Vacuum (From Greek age to XIX century)

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Abstract

A review of the History of the development of Vacuum Science and Technology is presented. The philosophy on structure of the matter in the Greek age from Anaxagoras to Aristotle. Alexandria. The discovery of the vacuum as absence of matter: Baliani, Galileo, Torricelli, Berti, Magni. The controversy, vacuist and plenist: The ether theory: Descartes, Newton, Leibniz. The discovery of real vacuum pumps: Otto von Guericke, Pascal, Huygens. The technology and science in the XVIII century. The new methods of vacuum production in the XIX and the greatest discoveries: electron, X-Ray. The theory of gases. The fall of the ether: Michelson-Morley and Einstein.

Keywords: Vacuum, History

Low temperature polysilicon TFTs on polyimide substrates for flexible electronics

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Abstract

In this work we present a novel process to fabricate low-temperature poly-silicon (LTPS) TFTs on flexible substrates. In particular, we fabricated non self-aligned LTPS TFTs on polyimide (PI) layers spin-coated on Si-wafer, used as rigid carrier, overcoming difficulties in handling flexible freestanding plastic substrates and eliminating the problem of plastic shrinkage with high processing temperatures. The device active layer was crystallized using excimer laser annealing while the gate oxide was deposited using electron cyclotron resonance PECVD. After TFTs fabrication, the PI layer is mechanically released from the Si-wafer and the devices have been electrically characterized under different bending conditions. In addition, hot carrier and self-heating related instabilities have been studied and analysed by using numerical simulations.

Simple circuits, such as inverts and ring oscillators, have been fabricated using the proposed process and tested.

Keywords: polycrystalline silicon TFTs, excimer laser annealing, self-heating

Structure and properties of sputtered Ag-Cu-O films: effect of the oxygen flow rate and of the target chemical composition

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Abstract

The ternary Ag-Cu-O system contains two defined compounds: $Ag_2Cu_2O_3$ and $AgCuO_2$. Recently, we have shown that the reactive sputtering process can successfully be used either to modify the metal stoichiometry in silver copper oxides or to deposit nanocomposite films (nc-Ag + nc-oxides) by adjusting the deposition conditions. In this presentation, we will discuss about the structure and the properties of Ag-Cu-O films as a function of the oxygen flow rate and the composition of the Ag-Cu target: either with eutectic composition ($Ag_{60}Cu_{40}$) or equiatomic ($Ag_{50}Cu_{50}$).

The film structure was determined by X-ray diffraction. The energy dispersive X-ray spectroscopy was used to estimate the film chemical composition. The film morphology was observed using scanning electron microscopy. The electrical and optical properties of Ag-Cu-O films were characterized using the four point probe method and UV-visible spectroscopy, respectively.

Whatever the composition of the target, the structure of the deposited films is strongly dependent on the oxygen flow rate introduced in the deposition chamber. Using the target with eutectic composition, the variation of the oxygen flow rate allows the deposition of the following structures: Ag-Cu-(O) solid solution, nc-Ag + nc-Cu_2O, nc-Ag + nc-$(Ag,Cu)_2O$ and finally X-ray amorphous. The occurrence of metallic silver into the sputtered films was confirmed by UV-visible reflectance measurements. On the other hand, the films deposited by sputtering of the equiatomic target with high oxygen flow rates exhibit the $Ag_2Cu_2O_3$ structure.

For both types of targets, the increase of the oxygen flow rate induces a continuous increase of the film oxygen concentration that can be correlated to the evolution of the deposition rate, the film reflectance and the film electrical resistivity. The occurrence of metallic silver is discussed as a function of the reactivity of metallic atoms versus oxygen.

Investigation on renal stone analysis with ToF-SIMS

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Abstract

Time-of-flight secondary ion mass spectrometry is being increasingly used for a variety of material investigation problems. This technique has a wide range of application in research and industry- failure analysis in manufacturing, semiconductors characterization, metals analysis, and the most challenging area of investigation by spectroscopic techniques, non-conducting materials. Its parallel detection of all elements, parent like molecules, fingerprint fragment ion formation from particular molecular compound and the charge compensation with low energy pulsed electron flood gun, makes it a valuable choice for non-conducting biomaterial analysis. The research community is expanding and progressing in interdisciplinary fields to explore new, best, precise and more informative techniques to replace or evaluate the old conventional techniques.

On the other hand, worldwide medical community is facing challenges in coping with renal stone disease. Up till present it is even difficult to stop its reoccurrence. The one main hindrance is non-precise chemical composition analysis with conventional techniques- like wet chemical analysis, which only gives information for selected compound analysis.

In this work, we successfully employed ToF-SIMS technique to solve the aforesaid problem because this technique gives information about all elements present in a specimen. In this preliminary stage we are acquiring fingerprint mass spectra from pure chemical compounds and identifying the characteristic fragments. These chemicals were acquired commercially in powder form. Samples were prepared by pressing powder on Indium sheets to make thick films to minimize substrate contamination effects. These specimens were analyzed for positive and negative ion spectra. The resulting database is then used to identify and characterize patient kidney calculi, recieved for chemical analysis on request from hospitals.

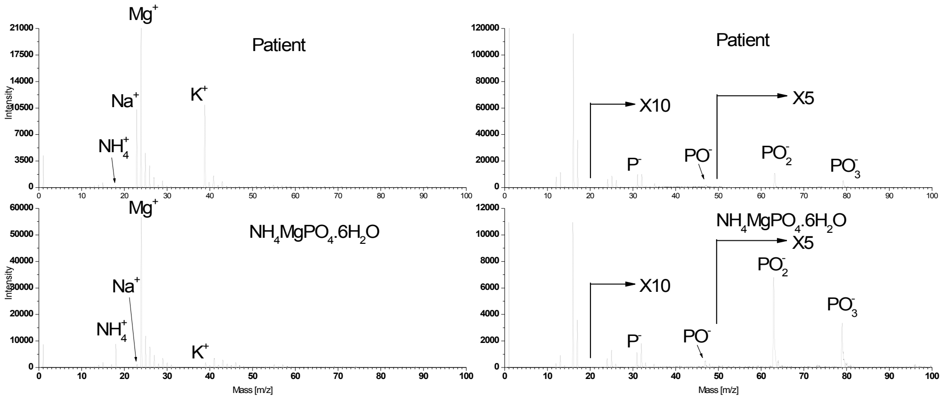

Fig. 1: Positive ion (left) and negative ion (right) ToF-SIMS spectra of patient kidney stone(top) and commercial chemical $NH_4MgPO_4.6H_2O$ (bottom) acquired at Ga^+ ion source energy of 12kV.

For instance, patient kidney calculi and reference chemical positive and negative ion spectra are shown in figure 1. Looking at the characteristic peaks present at 18 (NH_4^+) and 24 (Mg^+) along with negative ion spectra characteristic peak at 31 (P^-), 47 (PO^-), 63 (PO_2^-) and 79 (PO_3^-) one can clearly say that it is struvite stone ($NH_4MgPO_4.6H_2O$) with some additional minerals like Na^+ and K^+.

Keywords: Renal stone, Kideny stone, ToF-SIMS.

Highly conductive p-type nanocrystalline silicon films deposited by rf-PECVD using silane and trimethylboron mixtures at high pressure

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Abstract

P-type nanocrystalline silicon (nc-Si:H) films are attractive for use in a-Si and nc-Si solar cell application, since it has very high electrical conductivity and it is stable under illumination (decreased degradation). High optical band-gap between 1.9-2.1 eV is desired to reduce optical absorption, in the p-layer.

Thrimethylboron (TMB) has been receiving attention as a better alternative to diborane and methane mixtures for the deposition of p-type silicon carbide films for application in solar cells. TMB is particularly well suited for solar process deposition applications because it has a superior thermal stability relative to the more conventional boron sources, and can obtain films with an optical gap 0.1 eV higher than with diborane, without reduction of electrical conductivity. In spite of this success, p-type nc-Si:H films are difficult to obtain using TMB.

P-type nc-Si:H films were obtained by 13.56 MHz plasma enhanced chemical deposition (PECVD) using high hydrogen dilutions (> 98%) and a much higher pressure (2 to 5 Torr) than is used in typical hydrogenated amorphous silicon (a-Si:H) deposition.

The films obtained were characterized by Spectroscopic Ellipsometry, Raman Spectroscopy, and electrical conductivity measurements.

We investigate the influence of deposition parameters, namely working pressure and plasma power density, on formation of highly conductive p-type nc-Si:H films with high bandgap. Dark conductivity of 7.5 S/cm was achieved for a thin film with the thickness of 130nm. Despite of a high crystalline fraction (94%) of this film, deduced from spectroscopic ellipsometry, the bandgap of the same film was ~2 eV, which makes this material a perfect candidate for a window layer in both nanocrystalline and amorphours Si solar cell.

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Interface properties of Al_2O_3 thin films deposited on c-Si by pulsed ultrasonic sprayed pyrolysis

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Abstract

The interface quality of aluminum oxide (Al_2O_3), thin films deposited on silicon has been studied by XPS, IR Spectroscopy, SEM, and Electrical measurements. The films were deposited at temperatures from 400 to 550ºC from a mist of $Al(acac)_3$ in N,N-DMF by pulsed ultrasonic sprayed pyrolysis, a parallel mist of H_2O-NH_4OH was supplied simultaneously during the deposition process. High quality films with good stoichiometry and excellent interface properties were obtained. XPS and IR measurements suggest the presence of a silicon oxide (SiO_2) layer at the silicon interface. This SiO_2 layer could play an important role in the electrical properties of the Metal-Oxide-Semiconductor (MOS) structures fabricated with these Al_2O_3 films. Capacitance-Voltage measurements were carried out on these MOS structures and it was measured an interface state density in the 1010 $eV^{-1}cm^{-2}$ range at midgap.

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High speed surface functionalization of woven and non woven textiles

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Abstract

Conventional functionalization of woven and non woven textiles using various “nano” treatments and coatings is performed by immersing fabrics in water and solvent based solutions, followed by drying and curing. A unique “dry” surface functionalization technology has been developed that does not require the use of water or solvent chemistry. Highly cross linked and durable functional nanothick polymer layers are bonded onto the fabric fibbers without altering the material breathability and feel. Multifunctional coatings that combine hydro and oleo phobicity with flame resistance, color and antibacterial properties, have been developed. Plasma pre-treatment is used in place of wet initiators, surfactants, and adhesion promoting compounds. The same fluoro acrylate compounds that are used today to treat apparel fabrics in wet baths are converted into vapor by flash evaporation, deposited conformally on the surface of the fabric fibbers at supersonic speed which allows complete or partial fabric penetration, and are cured by electron beam radiation. This new dry fabric finishing process requires a small fraction of the electric power and real estate utilized by conventional wet finishing processes. The presentation describes the new fabric finishing process and presents data on fabric coatings with different surface functionalities.

Keywords: vacuum coating; high-speed coating; nanolayers; textiles

Sputtered Carbon coating Characterization and Bias voltage effect

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Abstract

The properties of sputtered a-Carbon and Cr doping a-Carbon carbon coatings on Si substrate using Closed Field Un-balanced Magnetron Sputtering Ion Plating (CFUBMSIP) deposition techniques were studied. XPS results clearly showed around 30-40% Sp3 involved in these a-Carbon coatings; this explained the reason of such hard carbon based coating. Scratch test results revealed that very thin Cr interlayer can produce very good adhesion with the Si substrate, but the higher Bias (60V) samples have less adhesion comparing to the lower bias samples (40V). The impact test and scratch tests shows the higher bias (60V) produced lower toughness and lower adhesion for both a-Carbon and Cr doping a-carbon coating due to the higher compressive stress inside the coatings. AFM results show very smooth film surfaces, the results show that using multi scratch method by applying constant sub critical load is a good method to characterize the toughness of the coating samples.

Physical properties of ultra fast deposited micro and nano thickness amorphous hydrogenated carbon films for medical devices and prostheses

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Abstract

Diamond like hydrogenated carbon films have been formed on different substrates at very low energies and temperatures by PECVD process employing acetylene as the precursor gas. The plasma source was of a cascaded arc type with Ar as carrier gas. The films grown at ultra fast rates were found a practical thickness limit of ~1.5 μm above which delamination from the substrate occurred.

Deposition on Si (100), glass and plastic substrates has been studied and the films characterized in terms of sp^3 content, roughness, hardness, adhesion and optical properties.

Deposition rates up to 20 nm/sec have been achieved at substrate temperatures below 100°C. The typical sp^3 content of 60-75% in the films was determined by XAES technique.

The hardness, reduced modulus and adhesion of the films was measured using a MicroMaterials Nano Test Indenter/Scratch tester. Hardness was found to vary from 4 to 13 GPa depending on the admixed acetylene flow and substrate temperature. The adhesion of the film to the substrate was significantly influenced by the substrate temperature and whether an in situ DC cleaning was employed prior to the deposition process.

Hydrogen content in the film was measured by a combination of FTIR and RBS techniques.

Adhesion problems related with deficient ion cleaning of the substrates

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Abstract

The up-scaling of the sputtering process from laboratory to industrial dimensions brings many times several problems not always completely identified. Probably, those which have required more concerns are the “adhesion failures”. For most of characterization techniques the adhesion of sputtered coatings achieved in laboratory developments is enough, reason why, researchers do not dedicate their efforts to the optimization of this property. However, from the moment that any coating reaches the interest to be implemented in the market, one of the challenges is the improvement and optimization of the coatings adhesion. Extensive studies of the influence of cleaning solvents, type of water, in-situ plasma surface erosion procedures and interlayers are carried out before the use in-service of the coatings.

In this work, a case of adhesion problems, occurred during the up-scaling of the deposition of tribological coatings in a semi-industrial apparatus, will be presented. A hard, from the W-Ti-N system, and a self-lubricant, of W-DLC type, coatings previously developed in laboratory equipments were deposited in a 4 cathodes TEER® chamber by reactive unbalanced magnetron sputtering. Besides normal ex-situ cleaning, prior to the deposition the substrates were ion bombarded by establishing a discharge close to the substrate, following the procedure usually adopted in industrial processes. Due to insufficient adhesion critical load values (measured by scratch-testing) a detailed analysis of the interfaces was performed in order to understand and overcome this problem.

A powerful set of complementary techniques were used to clarify this unexpected behavior: RBS permitted to identify the nature of the problem by detecting composition anomalies in the interface substrate / coating; Auger spectroscopy was used for identifying the chemical composition in-depth close to the interface; cross section TEM gave the final evidence of the presence of a contamination layer in that interface attributed to malfunctioning of the ion cleaning process.

Keywords: Adhesion, Coatings, Sputtering, Plasma cleaning

Synthesis and structural properties of W–Se–C coatings deposited by PVD in reactive and non-reactive processes

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Abstract

Transition metal dichalcogenides (TMD) are well known for their self lubricant properties, due to unique crystal structure. However, TMD materials present some drawbacks, such as, their low hardness which makes them inappropriate in applications requiring high load bearing capacity. This problem has been partially overcome by alloying or doping them with metals, carbon, or nitrides, solution applied with success on MoS_2 and WS_2 based coatings. Nevertheless, there is still a considerable potential for further improvements since others TMDs such as disselenides remains almost unknown.

W-Se-C coatings were deposited by sputtering from a WSe_2 target. The C content was varied from 0 at. % up to 70 at. % by either co-sputtering from a C target with different applied powers or by working in reactive mode in an Ar + CH_4 atmosphere. In the last case, the different carbon contents were achieved by varying the fluxes of the gases, maintaining the same total pressure for all depositions. The Se/W ratio during co-sputtering from two individual targets was between 0.9-1.0, with no significant influence of the carbon content, while in the reactive process it reached values higher than 1.7.

The hardness of the coatings was evaluated by nanoindentation with conditions that eliminates the influence of the substrate, with values between 0.6 - 5.6 GPa. The main influence for the hardness is the Se/W ratio, with values higher than 3 GPa for films with Se/W lower than 1.0 regardless the C content; for Se/W higher than 1.0 the hardness is between 2.5 – 0.6 GPa. In this case, increasing C content led to progressive improvement in the hardness.

The structure of the coatings was analysed X-ray diffraction in glancing mode, showing that the coatings have an amorphous structure.

Keywords: Transition metal dichalcogenides (TMD), WSe_2, Carbon based coatings

Protective YSZ-based thin films deposited by RF magnetron sputtering

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Abstract

The protective Cu-free and Cu-containing $Zr(Y)O_{2-\delta}$ films sputter-deposited onto apatite-type $La_{9.83}Si_{4.5}Al_{1.5}O_{26}$ (LSAO) ceramics were appraised for potential applications in solid oxide fuel cells (SOFCs) with silicate-based solid electrolytes, where the performance may suffer from the surface decomposition processes in reducing atmospheres. The crack-free, dense coatings with thickness of approximately 1 µm were applied using radio-frequency (RF) magnetron sputtering from a 99.95% pure yttria-stabilized zirconia target. Whilst as-prepared $Zr(Y)O_{2-\delta}$ films without copper additive were already crystallized and single-phase, fresh Cu-containing $Zr(Y)O_{2-\delta}$ are essentially amorphous, requiring high-temperature treatment in air for the crystallization of fluorite-type solid solution and CuO phases. On the basis of microstructural analysis and profile measurements, the deposition rate of 0.50-0.75 µm/hour at the sputtering power of 300 W was selected. The surface morphology studies using an atomic force microscope (AFM) revealed typical film structures with small (< 50 nm) grains. The hardness of these films, close to literature data on similar compositions, decreases from 15.8 to 8.4 GPa with increasing copper content. The anodic overpotential dependencies on the current density were analyzed by the 3-electrode technique and impedance spectroscopy, with porous Pt counter and reference electrodes, in 10%H_2-H_2O-N_2 atmospheres. The polarization of model electrochemical cells with cermet anodes comprising Ni, yttria-stabilized zirconia and $Ce_{0.8}Gd_{0.2}O_{2-\delta}$ (50:30:20 wt.%), was found essentially unaffected by the protective zirconia films, thus confirming applicability of this approach for the development of fuel cells with silicate electrolytes.

Keywords: yttria-stabilized zirconia (YSZ), magnetron sputtering, thin film, solid oxide fuel cell (SOFC)

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Advanced characterisation of nanometer multilayers: depth profiling comparison of RBS, SIMS, XPS and GDOES

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Abstract

The use of nanometre metal and/or metal compound multilayer coatings is extensively spread in a wide range of applications (i.e. hard protective coatings for mechanical parts and tools, optical coatings for lenses and architecture glass panels, barrier contacts for microelectronics, biomedical prosthesis, etc.). In these and other applications, sharp interfaces and a low degree of mixing between the component materials are strictly required. Obviously, the attainment of these complex structures needs of high resolution analytical techniques to get information about surface and depth composition at nanometric level.

In this work, the range of application of complementary analysis of nanometre multilayer coatings applying Rutherford backscattering spectrometry (RBS), Secondary Ion Mass Spectroscopy (SIMS), X-ray Photoelectron Spectroscopy (XPS) and Glow Discharge Optical Emission Spectroscopy (GDOES) techniques will be discussed. Each of the techniques has several pros and cons: RBS is a non-destructive technique that requires no standard for quantification although the access to large ion-source facilities is needed. SIMS keeps nanometre resolution values at larger depths with excellent lateral resolution, but at the expenses of large experimental times. XPS either using angle resolved measurements or simultaneous ion bombardment provides the depth profile together with chemical information although such information can be modified by the ion bombardment process. Finally, GDOES allows depth profiling in a fast and accurate way, even though depth resolution degrades linearly with depth due to the sputtering crater shape.

In particular, we have compared the depth profiling performance of the different techniques to the case of Cr/Ti multilayer structures with individual thickness ranging from hundreds to a few nanometres. Ultra thin chromium layers of 2.5 and 5 nm, buried at different depths in a titanium matrix up to a thickness of 3 µm, were properly resolved both near the surface and deeply embedded in the matrix and used to evaluate the depth resolution of the techniques.

Keywords: Multilayer coating, depth profiling, surface analysis techniques

Superhard nanostructured coatings: structure and properties relations

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Abstract

Nanocomposite thin films consisting of both nano-size solid solutions and nano-size polycrystalline materials embedded in various amorphous matrix materials provide a great potential for mechanical devices. Special attention will be dedicated to Ti-Si-N and Ti-Si-Al-N films prepared under conditions out of thermodynamic equilibrium, and the formation of mixed phases. In order to explain the influence of Si → Ti substitution in the bulk stoichiometric B1 TiN, calculations using first principles density functional theory were performed. A 16-atomic supercell Ti_7SiN_8, which corresponds to the nominal composition of $Ti_{0.875}Si_{0.125}N$ was used as a model. A lattice compression in result of Si-Ti substitution was obtained, which explains the experimental results.

These results suggest that, when surface mobility is reduced, the subsequent formation mechanism of nanocomposite of the type nc-MeN/a-Si_3N_4, results from the ion irradiation induced effects and a driven Si surface segregation, leading to continuous renucleation during film growth, which occurs in competition with the spinodal phase segregation.

This mechanism explains the grain size behaviour together with lattice parameter and the formation of a multiphase system, where the crystalline TiN, (Ti,Al)N and (Ti,Al,Si)N phases were identified by X-ray diffraction, when the surface mobility is not enough to obtain a complete thermodynamically driven segregation. XPS measurements show the absence of Titanium Silicide and the formation of the amorphous Silicon Nitride phase. These results show that a multiphase system of the type nc-TiN/nc-(Ti,Al)N/nc-(Ti,Al,Si)N/a-Si_3N_4 can be formed. This multiphase system revealed a formation of thermally very stable superhard nanostructure, with a significant hardness increase with annealing at 800ºC, which was retained up to 1000ºC.

Keywords: superhard coatings, nanocomposite, two-phase, Ti-Al-Si-N

Synthesis and Properties of Silicon-Aluminium Based Nanocomposite Coatings

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Abstract

Nanocomposite coatings consisting of an amorphous or crystalline short range order Si_3N_4 matrix and crystalline TiN or TiAlN phases are well known in the technical literature [1-5]. These coatings provide good abrasive wear resistance and surface chemical inertness of cutting tools and duty parts for engines, transmissions, and a large variety of other mechanical elements working under high load and/or extreme temperature.

This paper deals with the synthesis of MN-AlN-Si_3N_4 coatings, being M a transition metal like titanium, chromium or zirconium. These coatings have been obtained by simultaneous reactive deposition of Si-Al and the transition metal, produced from two cathodic arc sources. They have been deposited on M2 high speed steel substrates, with total thickness varying between 1.5 and 5.5 µm.

Deep profile analysis of the chemical composition of these coatings have been obtained using the Glow Discharge Optical Emission Spectroscopy (GDOES). MN-AlN-Si_3N_4 coatings typically exhibit 16-19 at.% of silicon and 10-18 at.% of aluminium. The percentage of the additional transition metal varies within the range 12-24 at.% and nitrogen is in all cases around 50 at.%. Microhardness of TiN-AlN-Si_3N_4 and CrN-AlN-Si_3N_4 coatings was found to be around 18 GPa, while hardness of the ZrN-AlN-Si_3N_4 is significantly lower, about 11-12 GPa.

The tribological performance of these coatings was evaluated thought reciprocating ball-on-disk experiments, where steel disks coated with MN-AlN-Si_3N_4 were tested against 100Cr6 steel balls. This kind of protective coatings based on the Si-Al system showed excellent anti-wear properties.

Keywords: Si-Al-N based hard coatings, nitrides, PVD arc

References:

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Monday Afternoon

7th Iberian Vacuum Meeting

5th European Topical Conference on Hard Coatings

Cooperative Molecular Field Effect-A Source for New Electronic Properties at Interfaces

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Abstract

New electronic and magnetic properties are induced by the adsorption of closed packed monolayers on solid substrates.[1] In layers made from chiral molecules, unexpectedly large electronic dichroism is observed, which manifests itself as spin specific electron transmission.[2] For many thiolated molecules self-assembled on gold, a surprisingly large paramagnetism is observed.[3] Self-assembled monolayers of double-stranded DNA oligomers on gold interact with polarized electrons similarly to a strong and oriented magnetic field. The direction of the field for right-handed DNA is away from the substrate. Moreover, the layer shows very high paramagnetic susceptibility. Interestingly, thiolated single-stranded DNA oligomers on gold do not show this effect.

All the observations can be rationalized by assuming orbital magnetism of the organic thin layer.[4] This is a new type of magnetism, induced by the formation of closed packed layer of organic molecules on metal. The adsorption results in charge transfer between the substrate and the adsorbed layer, which is the origin of this magnetism.

Key words: Interfaces, monolayers, field-effect

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Planck cryogenic testing in CSL premises, Helium partial pressure management.

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Abstract

Both satellite Planck and Herschel are cryogenic ones, the first one having a cold point around 0.1 [K], the second one around 0.3 [K]. Not only the detectors are cooled, but also major subsystems and systems of the spacecrafts.

The Centre Spatial de Liège (CSL) is involved in the testing of several parts of these spacecrafts, starting form optical tests on the mirrors or on the telescope, via cryogenic vibration testing of scientific focal plane instruments, ending with the full Planck spacecraft testing. Each test requires temperature lower than 20 [K], in volumes ranging from 1 [m^3] to 60 [m^3], cooling several kilograms to more than one ton, and withstanding heat load up to 150 [W] in stabilisation.

The overall Planck spacecraft test challenge is very high, as it is the only way to measure the end-to-end cooling chain of the spacecraft. The space conditions reproduction must be as perfect as possible to avoid the test set-up influence on the spacecraft performances, especially linked to radiative cooling, mechanical perturbations and Helium residual pressure.

Different challenges are presented, and the related CSL solutions are described, highlighting the Helium partial pressure problem, the related computations and trapping system by large sorption panel.

Keywords: cryogenics, thermo-optical testing, sorption.

Precise flow measurement of arbitrary gas species over a wide range

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Abstract

Numerous applications of vacuum technology require the precise measurement of gas flow. Challenges are very small flows in UHV experiments and sensitive leak detection, very large flows in tests of powerful coarse-vacuum pumps, and the use of exotic gases in plasma-coating and semiconductor processing. One basic approach for gas measurement is the usage of constrictions with known properties (conductance) which reduces the problem of flow measurement to the problem of pressure measurement. Various types of constrictions may be employed, i.e. the thin orifice, the Venturi-nozzle, and capillary-like devices.

The flow through the various constrictions depends on type and dimensions of the constriction as well as on the experimental conditions. This may be illustrated by examples: In case of an orifice, the throughput in the molecular regime is proportional to the thermal molecular velocity of the gas, and in the viscous regime it additionally depends on the isentropic exponent. In case of a capillary, the throughput is determined by the viscosity of the gas species. In the talk, the behaviour of gas flowing through the various constrictions and the theoretical calculation of throughput is sketched. This fundamental approach provides insight in the important flow properties and to derive general scaling laws.

There is a strong need for precise experimental investigations of the characteristics of constrictions. The need stems essentially from two motivations:

- Testing the fundamental theoretical models which are used in the ab-initio calculations. Preferably, the corresponding measurements are performed on devices with perfect shape and well-known mechanical dimensions in order to obtain a decisive quantitative comparison of theory and experiment.
- Precise determination of the conductance of individual devices which are employed for precise measurements of gas throughput in practical applications. Frequently, these devices do not have perfect shape, their surfaces are not perfectly smooth, and their actual dimensions are not known with sufficient accuracy. Furthermore, the actual flow may differ from the simplified ideal flow behaviour assumed in the calculations.

In experimental investigations of gas flow through a constriction, the flow can be precisely measured by two fundamental methods: First, measuring the gas volume (at known pressure and temperature) passing per time interval through a duct. Second, measuring the pressure change in a vessel (with known volume) caused by gas flowing into or out of the vessel. In the talk, different technical realizations of these methods are shown and their performances are discussed. An analysis of direct and indirect error sources and the accuracy is performed.

Keywords: gas flow measurement, orifice, Venturi-tube, metrology

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H_2 and Ne Gas Gap Heat Switches

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Abstract

The solid surfaces are able to retain atoms and molecules (adsorption) and this ability increases tremendously at cryogenic temperatures (lower than 200K). The use of high specific surface materials (area/volume) enhances this phenomenon and allows to use it for technological applications [1] as for example the "cryopumps" where few grams of activated charcoal ($\approx$1000 m^2/gram) are cooled down to 20K allowing a fast and efficient pumping.

In this presentation, it is described how the adsorption at low temperature can be used to build cryogenic heat switches, devices offering the possibility to make or break thermal contact between two parts of a cryogenic system [2,3]. The ON (conducting) and OFF (insulating) states of the switch are obtained by varying the gas pressure between two copper blocks separated by 100μm ("gap"). This pressure is managed by acting upon the temperature of a small cryopump (activated charcoal) connected to the gap space and (loosely) thermally coupled to one of the copper block. For a "high" cryopump temperature, the gas previously adsorbed in the cryopump is released to the gap between the two blocks, allowing a good thermal conduction through the gas (ON state). On the opposite, cooling the cryopump allows a very good vacuum between the copper blocks breaking efficiently their thermal contact (OFF state). Experimental thermal characteristics (Conductance in the ON and OFF state, ON-OFF switching temperature) of such a "Gas Gap Heat Switch" are described using Hydrogen or Neon as exchange gas and are compared to theoretical calculations.

The heat switch type described here is mechanically simple and does not need moving parts at low temperature: this explain why, nowadays, it is widely used in thermal buses of satellites.

Keywords: Cryopumping, Adsorption, Heat Switch

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ALBA Ultra High Vacuum System status

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Abstract

ALBA will be a third generation synchrotron light facility to be built near Barcelona (Spain). Commissioning of the storage ring is foreseen to start at the end of 2009. The design phase of ALBA is completed, the production phase is in full development and the first components are being installed. A 100 MeV LINAC will inject electrons into a nominal energy booster synchrotron of similar circumference as the storage ring, so that both accelerators will share the same tunnel. The storage ring, working at 3 GeV with a circumference of 268.8 m, has been designed for a maximum current of 400 mA. The lattice is based on an extended DBA structure and has a nominal emittance of 4 nm.rad. The machine has a four fold symmetry with 4 long straight sections (8 m), 12 medium (4.2 m) and 8 short (2.6 m).

The storage ring will be divided into 16 vacuum sections of two types: the unit cell and the matching cell. The sections will be separated by sector gate valves; see figure 3. The vacuum chamber will be made of stainless steel with an electron beam vertical aperture of 28mm and horizontal aperture of 72mm with a slot 10mm high and 20mm wide to connect the vacuum chamber to the antechamber where crotch absorbers distributed all along the circumference will absorb the unwanted radiation. The maximum power density (with normal incidence) is 250W/mm^2.

The pumping will be by lumped ion pumps with addition of titanium sublimation pumps close to the main absorbers. The overall pumping speed is around 60000 l/s; this will maintain an average dynamic pressure of 1.10^{-9} mbar to achieve a beam lifetime of 10 hours at the designed current after the commissioning of the machine. No in-situ bake out is foreseen for the vacuum vessels

Transition metal dichalcogenides alloyed with carbon: a new class of self-lubricant coatings

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Abstract

The wide family of transitional metal dichalcogenides (TMD) offers extremely low friction coefficients in dry sliding, and negligible wear. However, such favorable parameters are strongly limited by ambient conditions. The tribological performance is excellent at vacuum or inert atmospheres, but it deteriorates at presence of oxygen and, particularly, water vapour. To prepare homogeneous TMD films, the magnetron sputtering is one the most convenient method; moreover, it allows easy alloying of TMD with other elements.

Among TMD family, only MoS_2 and to some extent WS_2 were studied systematically, while other candidates, such us selenides or tellurides, stood partially aside. Our preliminary results showed that molybdenum diselenide was much less sensitive to air humidity than corresponding sulphide, and that WS_2 co-sputtered with carbon significantly improved tribological properties compared to pure WS_2. Therefore, we have decided to join both studies and deposit Mo-Se-C coating, which was expected to overcome humidity limitation.

The Mo-Se-C coatings were prepared by r.f. magnetron sputtering with carbon content from 29 to 70 at.% and hardness increasing with carbon content to a maximum value of 5 GPa. The coating microstructure of selected samples was investigated by transition electron microscopy (TEM) showing randomly oriented platelets of $MoSe_2$ embedded in amorphous carbon matrix. The friction of Mo-Se-C coatings considerably decreased with increasing contact pressure to values as low as 0.02 at humid air and the coatings withstood long-term tests with hundreds of thousands cycles on pin-on-disc. The wear track characterization revealed the formation of pure $MoSe_2$ thin tribolayer on the surfaces in the contact influencing tribological behaviour and progressive reorientation of $MoSe_2$ platelets, which is considered to be dominant feature driving sliding process.

High Power Impulse Magnetron Sputtering; An overview of history, properties and current status

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Abstract

In plasma-based deposition processing, the importance of low-energy ion bombardment during thin film growth can hardly be exaggerated. Ion bombardment is an important physical tool available to materials scientists in the design of new materials and new structures. Glow discharges and in particular, the magnetron sputtering discharges have this advantage that ions present in the plasma are abundantly available to the deposition process. However, the chemistry at the substrate is usually dominated by the ions of the inert sputtering gas while ions of the sputtered material are rare. Over the years, various ionized sputtering techniques have appeared that can achieve a high degree of ionization of the sputtered atoms, often up to 50% and in some cases even as much as approximately 90%. This opens a completely new perspective in the engineering and design of new thin film materials. High power impulse magnetron sputtering (HIPIMS or HPPMS) is a relatively newly developed technique, which relies on the creation of a dense plasma in front of the sputtering target to produce a large fraction of ions of the sputtered material. In HIPIMS, high power pulses with a length of ~100 µs are applied to a conventional planar magnetron. The highly energetic nature of the discharge, which can involve peak power densities of several kW/cm^2, creates a dense plasma in front of the target, which allows for a large fraction of the sputtered material to be ionized. HIPIMS was introduced by Vladimir Kouznetsov in the mid 90's (see for example US patent 6296742), and has since then been investigated by an increasing number of academic groups. The technology is now taking its first steps into industrial applications. This presentation gives an overview of the history, physical properties and current industrial status of HIPIMS.

Keywords: HIPIMS, HPPMS, Ionized PVD

PVD Nitride Hard Coatings Production on Chromium Based Tool Steel Substrates

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Abstract

Scaling-up process for coating technologies from basic research to large scale applications, implies exhaustive studies regarding the product development for future mass production. This process includes factors such as the right selection of substrates (preferably for high demand applications), the coatings that best match the substrate properties and a wide knowledge about the process conditions and coating deposition techniques effect on the final product. With this purpose a set of samples were prepared based on chromium based tool steel alloys (widely used steels for metal mechanical applications) such as AISI H-13 and AISI-4140 due to its good mechanical performance even after long exposure at high working temperatures and AISI D-3 designed for high resistance to wear or to abrasion and for resistance to heavy pressure. Then a set of coatings were deposited based on IV, V and VI group materials in the form of nitrides, chosen to be applied as coatings such as TiAlVN, ZrN and CrN; by using available coating production techniques like Plasma Assisted Physical Vapor Deposition (PAPVD) and DC-Reactive Magnetron Sputtering.

Finally with the purpose of having the optimal settings for mass production, analysis on the coatings were conducted through film characterization techniques such as X-Ray Diffraction (XRD), X-ray Photoelectron Spectroscopy (XPS), Scanning Electron Microscope (SEM), micro and nanoindentation, wear and adhesion test in order to study the effect of process variables like substrate temperature, bias voltage and N_2/Ar ratio over intrinsic material properties such as crystal structure, film texture, strain micro deformation and mechanical properties like hardness, wear resistance friction coefficient and adhesion of the coating to the substrate.

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Hard coatings produced by plasma processes – technology and recent applications

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Abstract

Due to economical and ecological aspects the reduction of friction and wear has a high impact on research and development of functional coatings.

A well established class of hard coatings are diamond like carbon coatings (DLC) and their metal containing derivatives Me-DLC prepared by plasma processes such as PACVD, DC magnetron sputtering and plasma activated evaporation. DLC coatings offer a high potential in realizing multifunctional properties, e.g. the combination of wear resistance and friction reduction with low adhesion, wettability, or multisensoric properties. Major application fields for DLC coatings are automotive components, mechanical engineering and tools.

The maximum hardness that can be achieved by traditional hard coatings like DLC is about 4000 HV. For higher hardness so called “superhard” coatings have to be used.

The hardest material available up to now is diamond. Polycrystalline diamond can be deposited fully automatically and highly productive on a large scale by hot filament CVD. Due to its excellent tribological, thermal and electrochemical properties CVD diamond coatings are well suited for a wide range of applications, e.g. cutting and forming tools, axial seal rings and other components with a high tribological load. Electrically conducting boron doped diamond can even be used for electrodes, e. g. for waste water treatment.

A very promising approach for new superhard materials is the development of cubic boron nitride coatings (cBN). cBN films are deposited by RF magnetron sputtering. The material offers several advantages: very high hardness, second only in hardness to diamond, high oxidation resistance and low affinity to iron - in contrast to diamond, and very high wear resistance. The mechanical-tribological characterization indicates an enormous potential of cBN coatings for tools.

Keywords: diamond like carbon coatings DLC, diamond, cubic boron nitride cBN, hard coatings

Tuesday Morning

7th Iberian Vacuum Meeting

5th European Topical Conference on Hard Coatings

Nanoscale science on metal oxide surfaces

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Abstract

Scanning probe methods have played a crucial role in allowing us to understand chemical and physical processes on oxide surfaces. This talk will highlight some important advances using three substrates as examples, namely $TiO_2(110)$, $CeO_2(111)$ and $Fe_3O_4(111)$. All three materials are important model catalysts, while TiO_2 has potential as a substrate for templating nanoscale circuits. Attention will first focus on imaging the water surface chemistry of $TiO_2(110)$ and $Fe_3O_4(111)$ as well as related reactions with O_2 and SO_2 for comparison with DFT calculations. Reactions with $TiO_2(110)$ are dominated by oxygen vacancies, while only one termination of $Fe_3O_4(111)$ reacts with water. The interaction of Pd with $CeO_2(111)$ will be used to provide a bridge between the surface chemistry and surface engineering parts of the talk. Here we are interested in the redox behaviour of the system, the results pointing to charge transfer from Pd to CeO_2. This is important in connection with the use of Pd/CeO_2 as a CO oxidation catalyst. The second part of the talk will examine the potential for $TiO_2(110)$ to self-assemble Pd, guide the substrate structure of TiO_2 through ultrathin film growth and be modified by electron beams. Using scanning tunnelling microscopy and X-ray photoelectron microscopy we show that it is possible to grow 1 μm long metallic wires of width 3 nm. Formation of reduced ultrathin films results in well defined shear plane structures that offer the potential to template linear structures. Electron beams are shown to form well defined reconstructed areas, with a mechanism that probably involves local heating.

Thin-film silicon MEMS

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Abstract

An overview of our work on microelectromechanical (MEMS) devices based on low-temperature thin film silicon as a structural material is presented. The talk will discuss aspects related to the fabrication, electromechanical characterization, and applications of thin film silicon MEMS.

The fabrication of thin film silicon MEMS is made using surface micromachining techniques. The use of low-temperature fabrication processes allows the fabrication of MEMS microbridge resonators on glass and on plastic (PET) substrates. The use of different sacrificial layers (photoresist or aluminum) allows the use of thin-film structural layers deposited at temperatures between room-temperature and 350 ºC.

The electromechanical characterization of the thin film MEMS devices emphasizes the characterization of their behavior as microresonators. The quality factor and the dissipation mechanisms in electrostatically-actuated microresonators based on these materials are studied from the point of view of materials properties and microstructure design. Designs allowing different vibrational modes such as flexural and torsional are developed to achieve high-Q resonators. The deflection of the structure in vacuum, air and water are discussed. The detection of the mechanical motion of the devices is mainly achieved by optical addressing. Sensitive detection of the mechanical motion using integrated magnetic sensors will be presented.

Applications of thin film MEMS devices as micromechanical sensors for environmental, chemical and biological detection will be discussed. Strategies for device functionalization and sensitivities to the attachment of biomolecules such as DNA and proteins will be presented. Limitations and challenges, as well as, other applications, such as passive 3D structures and actuators will be presented.

Keywords: thin-film silicon, MEMS

Ultra-Sensitive Shape Sensor Test Structures Based On Piezo-Resistive Doped Nanocrystalline Silicon

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Abstract

This paper describes the miniaturization of dense piezoresistive sensor arrays based on doped hydrogenated nanocrystalline silicon (nc-Si:H) thin films deposited on flexible substrates. The substrates are 125 and 15 µm-thick polyimide foils that are rugged, flexible and bendable. The nc-Si:H films, with a thickness ~100 nm, are prepared by hot-wire and radio-frequency plasma-enhanced chemical vapor deposition under high-vacuum conditions. Longitudinal GF = -32.2 and +26 were obtained in n- and p-type films, respectively, with optimized doping level and crystalline fraction.

Sensors were fabricated on 15 µm-thick polyimide film for later incorporation in devices for medical applications. Six of those sensors were cut and used in a test structure with the shape of a cross to track the movement of the head of a bedridden patient. The 15 µm-thick sensors were glued along two 125 µm-thick polyimide strips forming the arms of the cross. This approach illustrates the concept of fabrication on a thin "skin-like" membrane followed by cutting and transfer to others surfaces. The concept will be further exploited in spatially dispersed sensing devices. The sensors, used in a Wheatstone quarter-bridge configuration, show similar sensitivity (output voltage ~30 µV/nm) when used either in the parallel or transverse relative directions of current flow and strain. The temperature dependence of the output voltage will be measured.

A further step towards sensing at the microscale was the fabrication of test structures using optical lithographic processes. The test structures consist of nc-Si:H piezoresistive rectangularly shaped elements, having lateral dimensions in the range 50-100 microns, made by reactive ion etching. Their piezoresistive response is analyzed in terms of reliability, sensitivity and linearity. The performance of the sensors is addressed by bringing them into contact to surfaces having specific topography and using them to map contours on these surfaces.

Keywords: piezoresistance, sensors, flexible electronics, thin films

Structure and self-organization of organic and bio molecules on single crystal Surfaces

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Abstract

Self-assembly and self-organization of molecules are the main strategies used in nature to permit life to emerge from its building blocks, and have inspired new trends in nanotechnology based on a bottom-up approach. Understanding the structure of molecular layers on surfaces is of a great importance because its atomic arrangement determines the mechanical properties, electronic behaviour and reactivity of surfaces. Therefore, the ability to control the surface structure at an atomic level is a crucial point both for advancing basic knowledge on these mechanisms and for designing new type of biosensors more specific and highly sensitive.

Surface science techniques present a large sensitivity that make them very useful to find out structure, chemical interaction and growth mode of molecular layers on surfaces. The kind of molecule-surface bonding is one of the key-points for the molecular structure. In this presentation we will review different molecular systems, based on STM images. Single layers of PTCDA molecules on single-crystal metal surfaces, as Au(111), are an example of a low-interacting system. By depositing some Fe adatoms we are able to modify the bonding and therefore, the layer structure. We can move from organic nanodots (0D) or molecular chains (1D) to 2D growth just by changing the experimental conditions[1].

Another interesting systems are the bio-molecules covalently bound to metallic surfaces through a thiol-motif [3]. Particularly we will see a basic case of cysteine on gold, and an application derived from it, as self-assembled layers of DNA and PNA on gold for biosensing applications. Diffusing cysteine molecules on the surface coexist with both, molecular islands lacking of internal order and with long ordered molecular rows. After a period of time, the long molecular rows evolve to develop ordered molecular networks of cysteine. With respect to DNA and PNA molecules, we look to correlate the structural properties of the layer with the biosensing activity. We have found that molecular orientation of PNAs strongly depends on surface coverage and the interaction with the substrate[2].

At last, we will see how the surface interaction can be used to catalyzed reactions as the dehydrogenation of polycyclic aromatic hydrocarbon to form fullerenes and other related materials. This reaction at surfaces emerges as a new methodology for a rational synthesis of fullerenes.

Keywords: Organic Molecules and surfaces

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The electronic properties of graphene

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Abstract

In this talk we shall review the electronic properties of graphene [1] and some of the recent developed applications. Graphene was discovered in the late 2004 at the Centre for Mesoscopic and Nanotechnology of the University of Manchester, directed by Prof. A. K. Geim. This two-dimensional material is made entirely out of carbon atoms which arrange themselves in a honeycomb (hexagonal) lattice.

The electronic density of this material corresponds to having one electron per each carbon atom. The lattice geometry and its corresponding symmetry group determines the form of the electronic energy dispersion associated with the electron Bloch waves. The interplay between the lattice geometry and the electronic content of the material put the Fermi energy in such a position that the Fermi surface is reduced to six points in the Brillouin zone. The electronic dispersion around these six points is observed to be isotropic and linear in momentum. A consequence is that the effective model that describes the electronic excitations around the Fermi points is given not by the Schrödinger equation but by the Dirac equation in two spatial dimensions. The two spinor elements describing the electron wave function around the Fermi points represents the fact that the honeycomb lattice has two atoms per unit cell.

The fact that the elementary excitations are effectively described by the Dirac equation has wide range and some times even unpredictable consequences on the electronic properties of this system. In particular, most of the results one reads in the condensed matter literature about the two-dimensional electron gas formed in the inversion layers does not apply to electrons in graphene. This means that thermodynamic, transport, and spectral properties of electrons in graphene need to be revisited. The prospects of finding new exciting physics in graphene, which in turn may lead to unexpected applications in nanotechnology has been fueling both the experimental and theoretical research in this rapidly accelerating field.

We shall review the transport properties of graphene and its bilayer and how these properties are a consequence of the Dirac-like nature of the spectrum of graphene.

Keywords: graphene, Dirac, nanotechnology

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Formation and thermal stability of monolayer and multilayer films by azidoacetonitile decomposition onto rhodium surfaces

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Abstract

Existence of two highly reactive functional groups on azidoacetonitrile molecule, N3CH2CN, provides an interesting possibility to study C-N chemistry on catalytic surfaces. In present work we have studied a surface reactivity and thermal evolution of azidoacetonitrile adsorbed on Rh (111) at low (100 K) and at room (300 K) temperature, as a function of surface coverage and temperature, using X-Ray Photoelectron Spectroscopy (XPS) and Low Energy Ion Spectroscopy (LEIS) which is sensitive to the topmost layer.

The experimental measurements were performed in a UHV multitechnique apparatus. The XPS spectra were taken using the MgKα source. The light beam of 1 keV He^+ ions were used in LEIS measurements in gentle condition to minimize cracking and desorption by using low beam currents and short exposure times.

The obtained experimental results show that Rh surface is highly reactive with N_3CH_2CN. Decomposition is observed even at low temperature adsorption by surface induced N-N bond breaking of azide group. At 300 K rhodium carbides, nitrides and cyanides were formed by dissociative adsorption of azidoacetonitrile. Thermal behaviour and stability of reaction products from surface and its decomposition via C-N bond scission is strongly correlated by the initial coverage of Rh (111).

From experimental point of view, the LEIS technique has shown to be highly sensitive to the first monolayer. Use of this technique as an analysis tool seems to be a promising way to study a reactivity of complex molecules with surfaces.

Keywords: adsorption, surface reaction, LEIS, XPS, azides.

Nanocrystalline diamond films: from growth to applications

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Abstract

Due to an unique combination of physical and chemical properties, such as extreme hardness, high thermal conductivity, high breakdown field, negative electron affinity, p-type doping, high acoustic wave velocity, wide optical and electrochemical windows, chemical inertness, biocompatibility, diamond is an excellent candidate for mechanical, optical, thermal, biomedical and electronic applications. Owing to their small grain size leading to low surface roughness and low friction coefficient, nanocrystalline diamond (NCD) films are well adapted for nanotechnological applications, micro- and nanoelectrical mechanical systems (MEMS, NEMS), surface acoustic wave (SAW) devices, as well as for conformal coatings. In order to fulfill the requirements of such applications, the film intrinsic properties and therefore the synthesis process have to be controlled.

The talk reports on the investigation of the microwave plasma assisted CVD process used for NCD film deposition from Ar/H_2/CH_4 feed gas. Both permanent and pulsed microwave discharges are considered. The use of a pulsed wave is mainly motivated by the existence of two additional degrees of freedom compared to the continuous wave regime: the pulse repetition rate and the duty cycle. Various growth conditions obtained by varying these parameters, gas composition, input microwave power, pressure or substrate temperature, are examined. Film characteristics such as surface morphology and topography, structural properties, microstructure and electrical properties are discussed using real-time in situ optical monitoring, SEM, AFM, visible and UV Raman spectroscopy, XRD, TEM, and electrical measurements. Spectroscopic measurements and plasma modeling are also carried out in order to investigate the plasma phase characteristics in growth conditions. Optimized elaboration conditions are employed for achieving very high frequency SAW devices based on AlN/NCD layered structures. Experimental results show that a frequency up to 4 GHz is obtained for layered structure exhibiting a phase velocity of 9200 m/s with a lithographic resolution of 600 nm.

Keywords: Nanocrystalline diamond, microwave plasma, surface acoustic wave devices

Amorphous carbon/Cr_3C_2 nanocomposite thin films

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Abstract

Amorphous carbon/Cr_3C_2 nanocomposite thin films with different carbon contents have been deposited by rf magnetron sputtering using a metallic Cr target and a mixture of Ar and CH_4 as process gas. The partial CH_4 partial pressure was varied between 0.07 y 0.12 Pa, about 15% of the total pressure. The substrate was negatively biased with bias voltages between 45 and 130V. Samples were deposited on polished Si substrates with thicknesses in the range 0.5-1.0µm. The carbon content measured by EDX and XPS analysis reveals that all the films have carbon contents higher than those corresponding to the Cr_3C_2 stoichiometry. The presence of the amorphous carbon phase was confirmed by both XPS and Raman analysis. The XRD indicates the presence of the Cr_3C_2 phase with a strong (002) preferred orientation. As the CH_4 partial pressure was increased, an increase of carbon content was observed from 49 to 58 at%, accompanied by an increase of the amorphous carbon Raman signal and a reduction of the Cr_3C_2 grain size from 18 to 9 nm. The films with higher carbon content showed very good tribological properties in the ball-on-disk test against alumina ball, with values of the friction coefficient and sliding wear rate as low as 0.16 and $3x10^{-15}$ $m^3/N\cdot m$, while keeping moderately high hardness values of 17 GPa.

Keywords: nanocomposite thin films, rf magnetron sputtering, cromium carbide, tribological properties.

How to design nanostructured PVD nitrides based coatings for increasing the durability of steel ?

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Abstract

The design of thin hard coatings at a nanometer scale seems to be very promising to improve the characteristics of coated parts. Unfortunately, most often only one specific property is really enhanced. Besides, the origin of such a "nanostructure effect" remains still not clearly elucidated, and a wider industrial development requires understanding the relationships linking structure and functionality.

The aim of this talk is to present how it is possible to control the films structure to achieve optimized performance in terms of tribological, mechanical, and physico-chemical behaviours. Both types of films architecture are studied, resulting either from a stratification of nanolayers, or from a nanodistribution of a crystallised phase into an amorphous matrix. To illustrate both structures, arc-evaporated nanomultilayered TiN/CrN and TiSiN nanocomposite coatings are more particularly developed, and compared to a TiN reference.

High wear resistance of TiN/CrN is explained by an original propagation mode of the cracks due to a fluctuating residual stress field, evidenced by TEM and synchrotron measurements [1]. Corrosion behaviour is greatly enhanced by an outstanding covering rate, and depends on the nature of the outer layer. An external CrN layer, affords an enhanced protection. The high oxidation resistance of TiSiN is attributable to the network of refractory SiNx, which acts as a diffusion barrier for oxygen and insulates TiN nanograins from the aggressive atmosphere [2]. An original in situ approach of the oxidation phenomena is proposed by means of Environment SEM.

Keywords: Wear, Oxidation, TEM, Nanostratified and nanocomposite films

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Semi-orthogonal turning of hardmetal with CVD diamond and commercial PCD at different cutting angles

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Abstract

Turning of abrasive hardmetal parts by chip removal is a new challenge for cutting tools, alternatively to grinding. In such applications, polycrystalline diamond (PCD) and polycrystalline cubic boron nitride (PCBN) are the most established market options. Chemical vapour deposited (CVD) diamond can be an excellent alternative considering that the absence of any binder phase and its higher hardness should allow its use for machining a wider range of hardmetal grades.

In this work, thick CVD diamond brazed inserts and commercial PCD cutting tools were used to dry turn WC-18wt%Co bars. The cutting parameters were: speed (s) 20m/min, depth (d) 0.2-0.4mm, feed (f) 0.1mm/rev for roughing operations; and (s) 60m/min, depth (d) 0.05mm, feed (f) 0.04mm/rev for finishing purposes. Two cutting angle configurations were used, taking the combination of the tool holder and the cutting insert: i) a positive insert with a 0° (neutral) rake angle (γ) and a 11° clearance angle (α); ii) a negative insert with γ=-6° (negative) and α=6°.

For the CVD diamond tools in roughing conditions, the results showed that the use of the highest clearance angle (α=11°) diminishes the cutting forces and the adhesion of the workpiece material to the flank face. In the case of the finishing operation, no sign of wear was detected for this angle configuration, and just a very small flank wear for the negative insert. The use of neutral rake angles diminishes the crater land in rough machining and prevents this wear mode in finishing operations. On the contrary, a regular crater land was formed when the negative cutting tool was used. In the case of the PCD ones, the cutting operation had to be stopped at half the cutting length achieved by the CVD diamond tools due to the very high tool wear that increased the cutting forces.

Keywords: Cutting tools; CVD diamond; hardmetal; cutting angle

Effect of air annealing on the structure and the hardness of Cr-Si-N coatings deposited using a hybrid arc-magnetron process

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Abstract

Since the middle of the 90's, the attention devoted TM-Si-N coatings (TM = transition metal) is still increasing. Among the different systems, Cr-Si-N presents attractive properties: high hardness and high oxidation resistance. This presentation aims to compare the behaviour of CrN- and Cr_2N-based coatings with various silicon contents when submitted to air annealing.

Cr-Si-N films were deposited on stainless steel substrates using a hybrid magnetron sputtering-arc evaporation process. The nitrogen flow rate introduced in the deposition chamber was adjusted to synthesise either CrN- or Cr_2N-based coatings. The silicon concentration in the films was fixed by the DC power applied to the silicon target. The films were characterised by X-ray diffraction, scanning electron microscopy, energy dispersive spectroscopy and sputtered neutral mass spectrometry. Oxidation tests were performed by air annealing at 700, 800 and 900 °C. The film hardness was measured by depth sensing indentation on as-deposited and oxidised films. For the last ones, the oxide layer was removed by polishing to estimate the hardness of the remaining Cr-Si-N coating.

Air annealing induces an oxidation of the coating and the oxide layer thickness is lowered by silicon addition. Whatever the silicon concentration, air annealing at 800 °C of CrN-based Cr-Si-N films induces a decrease of the film hardness close to 10 GPa. XRD and SNMS analyses show that CrN transforms into Cr_2N leading to the formation of voids that explain the hardness decrease. In addition to this effect, a silicon depletion is noticed in the Cr-Si-N film. On the other hand, Cr_2N-based Cr-Si-N coatings exhibit a better behaviour after air annealing due to the absence of structural transformation in the remaining film. Nevertheless, diffusion of silicon towards the steel substrate is still evidenced by SNMS. The use of a diffusion barrier layer lowers this effect.

Tuesday Afternoom

Poster Session

7th Iberian Vacuum Meeting

5th European Topical Conference on Hard Coatings

About The Investigation of the Solid Surface Structure Ingomogeneities of Compact Niobium Pellets

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Abstract

This report presents a method for investigating the solid porous surface inhomogeneities. It allows to extend the small-angle X-ray scattering (SAXS) and to increase the reliability of the results obtained, by the “combined” supplementing the SAXS with Hg porosimetry data.

The method proposed in this work was used to investigate the surface of compact Nb pellets obtained by high temperature vacuum sintering of niobium powder. This object allows to vividly demonstrate the possibilities of the “combined” method for solving the problems of surface physics. At the same time this pellets are widely used in electronic engineering as metal plates in oxide-semiconductor capacitors, which realize electrical capacity is an “Nb-anodic Nb_2O_5 – solid state electrolyte” metal-oxide semiconductor Nb-MDS system [1].

The technique described in [2] was used for the SAXS investigation. In distinction to the SAXS, which detects the submicropore of any mode, Hg porosimetry method detects only open pores. Experiments have shown that that most of submicropore are open, i.e. usage of Hg porosimetry results is quite reasonable to supplement the SAXS.

The ‘combined” application of the SAXS and the Hg porosimetry method to investigate the structural surface inhomogeneities has allowed not only choose the most appropriate approximation of surface inhomogeneities shapes (according to the SAXS data), but also to take into consideration the polimodality of the investigated submicropore system by revealing (on the basis of the Hg porosimetry) correlation between the shape and the radius of the pores.

Keywords: X-ray scattering, porosimetry, submicropores

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Surface study of cerium based oxides coatings obtained by cathodic electrodeposition on zinc

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Abstract

A surface study of electrodeposited cerium oxide based coatings on zinc substrates by using surface analytical techniques is presented. In a first step, X-ray Photoelectron Spectroscopy (XPS) was used to determine the surface chemistry and composition. From the analysis of the Ce 3d core level peaks, the amount of Ce (III) and Ce (IV) species present on the coating surface are calculated. Analysis in depth has been performed by Ar^+ sputtering to establish the evolution of both species from the top surface to inner parts of the coating. On the other hand, the analysis of the O 1s core level peak revealed a triple structure, where the contribution at higher binding energies (~533 eV) is subject of controversy in the literature. This structure has been attributed either to a defective cerium oxide, to the incorporation of hydroxyl groups, to contamination or, most commonly, due to adsorbed water. Upon sputtering, this oxygen contribution is still present at deeper areas of the deposit, probably due to the porous nature of these layers. Electron stimulated desorption (ESD) and Auger electron spectroscopy were performed in order to find out whether this contribution is a consequence of water adsorption. These results are compared with XPS measurements performed on the same sample after the electron bombardment of the surface.

Keywords: Cerium oxides, XPS, ESD, AES

An investigation of the dependence of nano-structural modifications and characteristics of Ti foil surface on N$^+$ ion energy

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Abstract

The surface modifications of Ti thin foil samples (50 μm) made by nitrogen ion implantation are studied by SEM, XRD, AFM, and SIMS. Nitrogen ions with 25 keV energy and fluence of 1 x 1018 N$^+$ cm^{-2} were implanted in Ti foil samples for 1600 seconds at different temperatures. XRD patterns showed the development of titanium nitrides of different compositions in the samples. AFM images showed clear changes in the granular structure of the Ti foil surface with increasing the temperature. The density of implanted nitrogen ions and the depth of nitrogen ion implantation in Ti foil studied by SIMS showed a minimum for N+ density as well as a minimum for penetration depth of N$^+$ ions in Ti at a certain temperature which are consistent with XRD results. Hence, showing a correlation between XRD and SIMS results. This phenomenon is explained on the basis of residual gas, substrate temperature, dissociation of water in the chamber and the gettering property of sputtered titanium.

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Chemical Surface and Electrical Characterizations of Membranes Modified with Room Temperature Ionic Liquids

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Abstract

Transport properties of porous and dense membranes can be modified by incorporation of substances able to favour/reject particular substances in a mixture, which gives the possibility of obtaining membranes for a specific separation. Particularly, membrane electrical modification can strongly affect the transport of electrolyte solutions and charged particles. Among other strategies, incorporation of room temperature ionic liquids (RTILs) into the membrane matrix can be of interest in different application of membrane processes [1-2].

This work is focused on chemical and electrical characterizations of modified porous membranes obtained by filling the pores with imidazolonium based RTILs by filtering them under vacuum; the ionic liquids used are: C_8MIMPF_6, C_4MIMPF_6 and $C_{10}MIMBF_4$. For comparison, a Nafion-C_8MIMPF_6 dense membrane was also studied. Chemical characterization of modified membranes was performed by x-ray photoelectron spectroscopy (XPS) and IRFT spectra analysis, while electrical measurements allow the determination of cation transport number in the membrane (t_{Na+}) obtained from membrane potential or electrical potential difference at both sides of a membrane separation two solution of the same electrolyte (NaCl) but different concentration.

Age effect on porous RTIL-modified membranes was checked by comparing both XPS and t_{Na+} values for fresh and old membranes, that means, samples maintained 4 years without special preservation. XPS results do not show significant differences, but a reduction of 20% approximately for Na^+ transport number in aged samples was obtained, which could be due to the partial lost of the RTLIs in the pores.

Acknowledgements: To CICYT (Project MAT2007-65065), Junta de Andalucía (Excellence Project P06-FQM-1764) and MEC (Acción Integrada Hispano-Portuguesa HP-2007-0004) for partial financial support.

Keywords: XPS, transport number, RTILs, membranes

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Effect of hydrogen concentration in the bulk of stainless steel on kinetics of tribodesorption

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Abstract

Friction of stainless steel in ultra high vacuum produces desorption of absorbed and occluded gases, among which hydrogen is the main component. This phenomenon is known as tribodesorption. Previous studies revealed the critical importance of the plastic deformation on the grove produced by friction on tribodesorption kinetics. When friction is performed in elastic regime, the tribodesorption rate is negligible. However, in the plastic regime, tribodesorption rate increases with increasing normal load. Another important topic, but not yet studied, is the effect of H concentration in the bulk on the tribodesorption rate. Therefore, the aim of the present study was to investigate the influence of the initial concentration of hydrogen in stainless steel samples on the tribodesorption rate.

Samples of AISI 304 steel were implanted with H using cathodic polarization in electrolyte solution of 0,1N H_2SO_4 with or without 2,5 g/l TeO_2 during 2 hours at room temperature. TeO_2 was used to enhance the H absorption. Current density was 10mA/cm_2. Hydrogen in depth distribution of both non-treated and H-implanted samples were performed using secondary ion mass spectrometry. Although this method does not allow absolute measurements without additional calibration, it provides useful information on the relative hydrogen concentration in the bulk of the samples. Tribodesorption measurements were performed in ultrahigh vacuum system. Sample surfaces were rubbed by an alumina ball under constant applied normal load of 0,2 N. Total amount of desorbed gases are determined from the p-t curves

For H-implanted samples without using Te the amount of desorbed gases increased by 83%, in respect to non-treated samples. This increase of the desorbed amount corresponds to the increase of the concentration of H in the bulk of 89%. For H-implanted samples using Te the concentration of H in the bulk increased by 266%, while the amount of desorbed gas decreased by 63% in respect to non-treated samples. This apparently contradictory finding, that at higher bulk concentration the tribodesorption rate is lower, can be explained by the formation of hydrides in the bulk.

Keywords: friction, hydrogen, gas content, stainless steel

Influence of the optical basicity on the photoemission yield of multicomponent glasses

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Abstract

The goal of this study is in spacecraft-plasma interactions, which deeply involves the insulation properties of outer coatings of a satellite. Photoemission experiments with VUV radiation have been performed to investigate the charging characteristics of different glasses by using a pulsed method. X-ray Fluorescence, Energy Dispersive X-ray and X- ray photoemission spectroscopy were used to obtain the glasses composition. The chemical stability of these glasses was studied by hydrolytic attack and also the ions lixiviated from the glasses were analyzed. The glasses were oxide alloys of a combination of acidic oxides (SiO_2, P_2O_6, …) and basic oxides (CaO, Na_2O, MgO, …); commercial glasses as pyrex, glass standard reference 1412, glass standard reference 89 or silice were also studied.

Glass particles of 2-20 µm diameter were exposed to a collimated beam of VUV radiation in the 110-200 nm spectral range from a deuterium lamp source with a MgF_2 window. Photoemission current implies positive charging of the surface of the exposed glasses. We have found that for thick glass samples (>1µm) the photoemission current decreases in function of time. In this case, a surface charge and corresponding surface voltage develop which hinder the photocurrent as they build up. When pulsed light is incident on the glass, the photoemitted electrons experience space charge effects between them and the positive charges on the surface. This Coulomb interaction gives rise to an abrupt photoemission current decrease. However, for very thin oxides the current is essentially constant. It is due to the photoinjection from the conductive substrate. The photoemission currents varied in the range 10^{-6} - 10^{-5} A/m^2.

The theoretical optical basicity of the glasses was calculated from their chemical composition. The optical basicity was found to vary in the range 0.48-0.66. Higher photoemission yields are obtained for the glasses with higher optical basicity.

Keywords: Photoemission yield, glasses, optical basicity, XRF

Complementing SIMS analysis with RBS and XPS

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Abstract

Secondary Ion Mass Spectrometry (SIMS) is a powerful surface analysis technique, with low detection limits and the capacity of detecting all elements, its isotopes and molecular aggregates. Only the top most atomic layers are probed by SIMS, depth analysis may obtained by sputtering the sample surface. However, this technique has the drawback of not being, by itself, absolutely quantitative, since the sputtering rate and the ionizing probability of the secondary (sputtered) particles depend on the surface composition (matrix effects) [1].

The matrix effects can be controlled in two ways: (1) using reference targets for comparison; (2) resorting to other analytical techniques.

In this work, we present a method that can be used to complement SIMS information, using two other analytical techniques: X-ray Photoelectron Spectroscopy (XPS) and Rutherford Backscattering Spectroscopy (RBS). During SIMS analysis, XPS is used to determine the elemental composition at selected sputtering steps. RBS, in turn, is used to determine the elemental depth distribution in the sample.

By comparing the atomic fractions yields measured by RBS and XPS, we obtain the depth at which the XPS (and SIMS) measurements were performed. By comparing XPS and SIMS results, we obtain the relation between the SIMS and the real (XPS) elemental fractions.

This approach was used to analyze an aluminium sample after a low energy, very high fluence implantation of $7Li^+$ ions (E = 10 keV, DN = 2.5×1018 $7Li^+/cm^2$), yielding consistent results [2].

Keywords: SIMS, RBS, XPS.

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Thermal Analysis Techniques in Pharmaceutical and Environmental Chemistry

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Abstract

Thermal analysis techniques are very useful in materials science where the properties of materials are studied as they change with temperature. Differential scanning calorimetry or DSC is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference are measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned. The basic principle underlying this technique is that, when the sample undergoes a physical transformation such as phase transitions, more (or less) heat will need to flow to it than the reference to maintain both at the same temperature. Whether more or less heat must flow to the sample depends on whether the process is exothermic or endothermic.

An alternative technique, which shares much in common with DSC, is differential thermal analysis (DTA). In this technique it is the heat flow to the sample and reference that remains the same rather than the temperature. When the sample and reference are heated identically phase changes and other thermal processes cause a difference in temperature between the sample and reference. Both DSC and DTA provide similar information; DSC is the more widely used of the two techniques. Those techniques are widely used in the pharmaceutical industry and also to study the behavior of some contaminants to prevent environmental problems. [1][2][3]

Keywords: Thermal analysis, drug, contaminant

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Tailoring the TiO_2(110) work function – Local or global effect?

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Abstract

Model systems for heterogeneous catalysis are widely studied, due to the expectation of gaining insight of its fundamental properties. One of such properties is the surface work function (WF).

Since the seminal work of Haruta on CO oxidation [1], supported gold clusters on titanium oxide have been widely studied. One of the key challenges is the correlation between structure and reactivity of the supported clusters. In previous works [2] we have shown that the shape and size of silver clusters grown on TiO_2 are important factors regarding the lowering of the surface work function.

In this work we show that the growth of gold clusters produces an increase of the surface work function and relate that change to cluster size and dispersion. The value of the TiO_2(110) work function falls in between the values of gold and of silver. By the choice of adsorbate, and growth conditions, it is possible to tailor the surface work function to a specific need.

Focusing on the sub-monolayer range, it is easily realized that due to clustering by the adsorbate, a portion of the original surface remains unchanged. Accordingly, it would be expected that the work function change only had a local effect; nevertheless the technique used to probe the work function, which tracks the onset energy of the secondary electron emission spectra and is sensitive to the work function minimum, points to a global change even when only 1/10th monolayer of adsorbate is present on the surface.

Some authors, using AFM and STM techniques [3-4] show interesting local effects from clusters on the surface work function. Is the surface work function change induced by adsorbates a local or global effect

Keywords: Work Function, Silver, Gold, TiO_2.

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Surface Analysis of EPDM Elastomer with Different Carbon Black Contents. Evolution after Tribological Tests

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Abstract

Surface analysis of peroxide-cured ethylene/ propylene/ diene elastomers (EPDM) was studied by X-ray Photoelectron Spectroscopy. This technique provide information about the surface chemistry of EPDM. The carbon black (CB; N550 type) content of the EPDM rubbers was varied between 0 and 60 parts per hundred parts rubber (phr). A comparison of the surface chemistry as a function of the carbon black content was performed. The results evidenced a small influence of the carbon black content on the surface composition of EPDM. In addition, the samples were tribotested against sliding steel counterparts under dry conditions in the Roller (steel)-on-Plate (rubber) configuration. The same XPS analysis were carried out inside the wear track in order to establish possible compositional changes after tribological tests. A smaller oxygen content was found inside the wear track on the samples with carbon black. The increasing percentage of carbon black reduces the presence of oxygen in the wear track. Finally, wetting experiments were also carried out by measuring the contact angle of water droplets on the EPDM surfaces using the sessile drop method. No effect of the carbon black content was found on the hydrophobicity of the EPDM. On the contrary, there is a clear tendency to increase the hydrophobic properties on the wear track of all the EPDM and specially in the sample without carbon black. The possible causes of these behaviour will be discussed.

Keywords: XPS, Elastomers, Carbon black, wettability

Biological samples preparation for oligoelements analysis by TOF-SIMS technique

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Abstract

The main objective of this work is to explore the potential of TOF SIMS technique for an experimental study regarding elemental composition of biological tissue, such as human hairs and nails, and to develop an experimental procedure compatible with the system requirements for ultra high vacuum.

An important step of this study was the sample preparation by using different protocols for an organic compounds reduction on these biological tissues.

Since these biological samples are insulators, we are successfully using the Secondary Ion Mass Spectrometry by Time of Flight (ToF-SIMS), which is a powerful analytical technique, suitable to investigate insulators, and allows detailed information about elemental and molecular composition of surfaces, including detection of all elements and isotopes.

In this work a powder samples, whole hair and nails were pressed on high level of purity Indium substrates and analyzed at room temperature by primary ion beam of Ga^+ of 12KeV in pulse mode. Charge compensation was achieved by low energy electron gun. Characterization of the chemical composition of different samples was performed by measurements in positive and negative mode SIMS spectra. Obtained experimental results shows that even reduced presence of organic compounds make difficult identification of some important for medical applications trace elements.

Keywords: TOF-SIMS, biological samples, oligoelements

Characterization of Aluminium-PMMA interface modified by Magnetron DC glow discharge

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Abstract

Polymer materials coated with thin metallic films are widely used in many domains of microelectronics and optics. In the scope of ALFA/ATLAS Forward Detectors for Luminosity Measurement and Monitoring, scintillating optical fibres are being coated with a high reflective aluminium thin film by DC magnetron sputtering. For this purpose reliable film adhesion is necessary without compromising optical properties at the coating/optical fibre interface.

In this work we present results from the XPS (X-ray Photoelectron Spectroscopy) analysis of the interface failure of aluminium films deposited on PMMA (Poly-methyl methacrylate, $C_5H_8O_2$), fibber cladding material. The films were deposited by Magnetron DC glow discharge using plasmas of Helium and Argon, without previous surface treatment.

The plasma deposition parameters for both gases where the same, 1 kW of power discharge at 5 Pa of work pressure. These plasma environments show remarkable differences regarding film adhesion to the substrate, due energetic sputtering particles affecting polymer surface. Samples deposited in He atmosphere show an increased adhesion strength of 1 to 15 KPa to values of 1 to 10 MPa, when compared with samples obtained in Ar atmosphere.

The samples are prepared by peeling the aluminium film and carrying the XPS analysis on the exposed substrate. The PMMA molecule has a low polar structure which leads to coatings with low adhesion strength. It can be seen in the XPS results that the adhesion was improved with polymer surface degradation, in particular when reducing the relative abundance of the bridging oxygen atoms (-C-O-CH_3) in sputtering process. Care was taken to avoid X-ray damage to the samples by operating the source at the lowest possible beam power.

Deposition of graphene by plasma enhanced CVD: an XPS study

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Abstract

Graphene is a promising material for fabrication of nanoelectronic devices. A possible way for its mass production is microwave plasma enhanced chemical vapour deposition (MW PECVD). The main advantage of this technique is that graphene flakes can be grown without the need of a catalyst on different substrates that can withstand high temperature (up to 700° C). However, the growth mechanism of PECVD synthesized graphene is not well understood. In this work the growth process of graphene deposited by MW PECVD with different parameters on several substrates was studied. Graphene layers formed on three different substrates (quartz, silicon, platinum) for different deposition times were characterized by angle resolved X-ray photoelectron spectroscopy (ARXPS). It was established that a carbide layer is initially formed on the Si surface followed by a deposition of an amorphous carbon film and growth of graphene. No intermediate carbide layer was formed on both quartz and platinum surfaces. Furthermore, no amorphous carbon layer was detected on the platinum substrates, even for the shortest deposition time intervals.

Keywords: graphene, PECVD, (AR)XPS

Theoretical Study of the Influence of Nanostructure on the Electric Properties of Polymer-based Diodes

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Abstract

Several experimental studies have shown that the nanostructure of polymer thin films used in organic diodes depends on the conditions used to deposit the polymer layer between the electrodes. The way that conjugated polymer chains are stacked and aligned relative to the electrodes, or their average length can influence the electric behaviour of polymer-based diodes, such as light-emitting diodes. By using a generalized dynamical Monte Carlo method we have study the influence of chain orientation at electrode-polymer interfaces on the charge injection process and charge mobility through the polymer layer in single-layer polymer diodes.

Keywords: Modelling, organic semiconductors, nanostructure

Metal / Metal Oxide Multilayer by Reactive Pulsed Laser Deposition

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Abstract

Multilayer coatings are used in a large number of various applications like data storage media, waveguides and X-ray mirrors. A wide range of materials can be used for the particular layer fabrication, which results in novel materials with advanced properties [1].

The mechanical behaviour (hardness, elasticity, and adhesion) of multilayer coatings is strongly influenced by the type of the formed interfaces between the different layers; and the interface region is predominantly not perfect, which is strongly dependent on the energetic conditions during deposition.

The current paper reports on the preparation and characterization of Zn/ZnO multilayer coatings by cyclic pulsed laser deposition (cyclic-PLD) technique. The advantages of this method arises mainly from: the high-kinetic energies of the laser ablated species that allows for a lower temperature growth, the use of high-purity precursors (Zn and O_2 gas), and the possibility to grow in a cyclic manner [2] and in the extreme flexibility of the deposition technique.

Metallic zinc targets were ablated with a Q-switched Nd:YAG laser, operating at 1064 nm wavelength with variable pulse energy of up to 100 mJ and pulse duration of 5 ns. Multilayer systems (single layer thickness d = 5 to 20 nm) have been deposited on Si (111) substrates by alternating vacuum and O_2 r.f. plasma cycles. Single layer thickness can be adjusted by controlling the vacuum and RF plasma cycle duration.

Structure and composition have been investigated by means of X-ray Reflectivity, High Resolution Transmission Electron Microscopy (HRTEM), and X-ray Photoelectron Spectroscopy (XPS).

X-ray Reflectivity scans in a θ–2θ mode usually show clear total thickness oscillations and sharp multilayer Bragg peaks indicating the presence of a well-ordered layered structure. High Resolution Transmission Electron Microscopy results show the extremely dense growth structures at the interface regions. XPS depth profile measurements show a regular structure of the layer sequence metal / metal oxide with a constant distance along the sample normal and sharply formed interfaces.

Keywords: Pulsed laser deposition, ZnO, Polycrystalline films

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Acknowledgements: This work was supported by Fundação para a Ciência e a Tecnologia (FCT).

ZnO Films Grown by Laser Ablation with and without Oxygen CVD

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Abstract

We have studied the properties of ZnO thin films grown by pulsed laser deposition (PLD) using a ZnO target under various background oxygen conditions and sapphire substrates. The substrate temperature was varied between RT and 800 ºC. The targets were ablated with either the IR line at 1064 nm or the 4th harmonic (266 nm) of a Nd:YAG laser.

Various methods are employed to obtain the optical, structural and electrical film quality, like optical transmission spectroscopy, X-ray diffraction analysis, RBS, SIMS and XPS for composition, SEM and AFM for surface analysis, photoluminescence, UV-excited photoconductivity, and time-resolved microwave conductivity. The X-ray analysis with rocking curves and θ-2θ scans were done with a Siemens D 5000 diffractometer with a copper source, indicating preferential growth in c-direction. The RBS analysis of the samples was performed at ITN with a 2.0 MeV He^+ beam, at normal incidence with standard and annular detectors (scattering angle 140º and 180º, respectively). Both film thickness and film composition was determined. We found that the composition of the films approaches stoichiometry with increasing film thickness.

Photocurrent measurements at RT with excitation by the 325 nm line of a HeCd laser showed a strong persistent photoconductivity effect (PPC) up several 100 seconds, indicating the presence of deep defects. The PPC signal showed smaller characteristic times for films having higher photo-to-dark ratio and better stoichiometry.

Our results obtained show that the relatively simple PLD technique is capable of producing ZnO films with good structural, electronic and optical quality. We are using the technique to get more insight into the problems of defect creation and of kinetics of charged impurities.

Acknowledgements: This work was supported by Fundação para a Ciência e a Tecnologia (FCT).

Keywords: Pulsed laser deposition, ZnO, Polycrystalline films

Multicrystalline silicon solar cells with a-Si:N:H antireflective coatings

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Abstract

Amorphous hydrogenated thin films are good materials for optoelectronic applications due to tuneable energy gap and refractive index. Exemplary film for this kind applying is silicon-nitrogen a-Si:N:H [1], obtained by PECVD method at 13.56 MHz. Deposition process occurs in following conditions: gaseous mixture $SiH_4 + NH_3$, with changeable NH_3 content in the plasma in the range 0.11÷0.3, deposition time 7–15 minutes, constant pressure 0.6 Tr and substrate temperature about 180°C. Borosilicate glass, monocrystalline silicon and multicrystalline silicon were used as the substrates. The process parameters were chosen to obtain the films of optimal energy gap, refractive index, and low effective reflectivity. Refractive index and energy gap of a-Si:N:H films were calculated from transmission and reflection measurements. The refractive index of the layer is in the range from 1.7 to 2.2, energy gap from 2.4 to 2.9 eV and the optimal film thickness d≈70 nm. The spectral dependence of the reflectivity of a-Si:N:H on both kind of silicon substrates have minimum values for wavelengths between 600 and 1000 nm. The optimal physical properties of films for this application were predicted by use simulation method based on two diodes solar cell model [2]. The measurements of solar cell electrical parameters by the use of computer controlled global spectrum sun simulator (I-V Curve Tracer For Solar Cells Qualification) indicated a significant increase in efficiency due to application of a-Si:N:H films. For improvement of solar cells electrical parameters also the passivation of defects by hydrogen is needed. FTIR analysis of a-Si:N:H films indicated presence numerous hydrogen bonds (Si-H and N-H), which passivates structure defects in multicrystalline silicon and reacts with impurities.

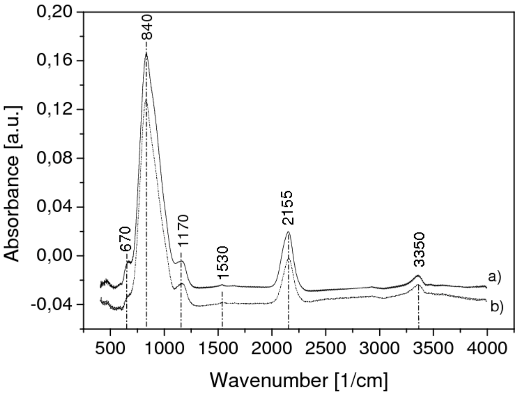

Fig.1. FTIR spectra of a-Si:N:H film obtained on Cz-Si substrates by PECVD for ammonia contents: a) wa= $NH_3/(NH_3+SiH_4)=0.11$, b) wa= $NH_3/(NH_3+SiH_4)=0.2$

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MF pulsed DC power supplies for PVD processes

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Abstract

Reactive processes, such as magnetron sputtering and cathodic arc evaporation take place in the hard, wear-resistant coatings deposition. Middle Frequency (MF) pulsed DC magnetron power supplies were firstly used since the late 80's to reduce or even prevent arcing in reactive magnetron sputter deposition of highly insulating layers due to the re-charging of the surface charges phenomena. Now pulsed bias power supplies prevent arcing onto the substrates during the cathodic arc evaporation processes.

We at Milko Angelov Consulting Co. Ltd. have more than ten years of experience in designing and manufacturing of variety of customer specified power supplies, both for magnetron sputtering and substrate biasing. Unipolar and asymmetric bipolar models are available. In this paper arc suppression and preventing during the deposition together with methods for increasing the ionization are discussed. Target poisoning, its role in the arc formation, deposition rate decreasing and the reason to prevent it by using of pulsed power supplies are further discussed. New approach of pulsed magnetron power supply for increased ionization is described. Pulsed sputter deposition equipment for deposition of a-Carbon protective and biocompatible layers onto cardiovascular stents is presented. Our own bipolar pulsed technology for substrate biasing is described.

All of our power supplies have built in fast arc suppression circuitry and state-of-the-art "Soft Arc Handling" facility, which reduces delivered energy in a single arc even at relatively low output currents, such as fifteen per cent of the maximal value.

We are known as a trusted and reliable manufacturer of MF pulsed power supplies, providing best "performance to price" ratio and reliability all over the world.

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Topic: Plasma Science and Technique.

Atmospheric plasma surface modification analysis by Energy Resolved Molecular Beam Mass Spectrometry and SIMS

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Abstract

Electrical plasmas can be readily produced at atmospheric pressure and have relatively low running costs. They are ideal for a variety of industrial process applications for many materials. Processing using non-thermal atmospheric plasma currently extends to areas such as surface cleaning and functionalisation, plasma activation, tissue engineering and sterilisation.

To aid in understanding the mechanisms involved in plasma/surface interaction we present results of both plasma measurements and surface composition studies for a range of materials treated using an atmospheric dielectric surface barrier discharge (DSBD). The plasma properties were determined using an energy-resolved molecular beam mass spectrometer (ERMBMS). The surface compositions of the materials before and after treatment were compared using a static SIMS instrument.

The dielectric surface barrier discharges were operated using helium gas and applied to molybdenum, silicon wafers and printed circuits. Silicon test pieces were treated using hydrofluoric acid to produce a strongly hydrophobic, hydrogen-terminated surface. The hydrophobicity of the surfaces could be significantly reduced by short exposure to certain plasma conditions. Static SIMS analyses of these surfaces showed a significant reduction in the observed SiH^+ signal and an accompanying increase in the Si and the reactive silanol groups [Si-OH].

ERMBMS consists of an energy mass spectrometer (QMS) with a differentially pumped three-stage inlet system [1]. Mass spectra of the ions generated in the DBSD source show a high concentration of monoatomic and diatomic oxygen. Analysis of the ion energy reveals that the ions are fully thermalized, with energies close to 0.03 eV.

ERMBMS analyses of the plasmas provided information on the relative contributions of ionic and radical species to the changes in the surface structure and the combination of data from the two diagnostic techniques contributes to our general understanding of such plasma/surface processing.

Keywords: energy mass spectrometer, dielectric surface barrier discharge, atmospheric plasmas, SIMS

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Real-time control of the ISTTOK hydrogen injection system

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Abstract

With the implementation of alternating discharges (AC) at the ISTTOK tokamak, the typical duration of the plasma discharges increased from 35 ms to 250 ms. In order to achieve longer discharges, a real-time control of the plasma fuelling is essential to balance the particle losses and the changes in recycling on the limiter and walls.

The injection system being used is operated by a piezo-electric valve in order to achieve precise puffing injections of hydrogen. However, if the amount of hydrogen injected is miscalculated or done out of time, the consequence is usually the quenching of the plasma. Therefore, it is necessary to establish a criterion for the precise quantity of hydrogen that should be injected.

In this paper, a real-time feedback control system based on a dsPIC embedded system is presented. The new control system evaluates data from the H-α radiation and crosses this information with a real-time plasma density calculation from the microwave interferometer data. The H-α radiation diagnostic is of extreme importance in this process because the amount of radiation detected is related to the number of particles being recycled near the walls and on the Tokamak limiter.

The digital signal processor transmits a pulse to the piezo-electric valve controlling the instant it opens and the duration, controlling in this way the amount of hydrogen injected. The system operates at a time scale of 1 ms, since this is the order of magnitude of the response delay of the piezo-electric valve.

The system is intended to operate regularly after an operational test being accomplished.

Keywords: Tokamak, active hydrogen injection, real-time control

Modeling of Capacitively Coupled Radio-Frequency Discharges in Nitrogen

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Abstract

Capacitively coupled radio-frequency (ccrf) discharges in nitrogen are increasingly used for plasma-assisted material processing applications. Unfortunately, there is only a partial knowledge about the behavior of nitrogen plasmas under ccrf discharge conditions, which limits reactor optimization (in terms of processing rates and uniformity), and justifies further modeling and experimental studies on the subject. The aim is to understand and control the strong coupling between the discharge operating conditions and the plasma parameters, resulting from the use of a very reactive processing medium such as nitrogen.

This work presents a characterization of 13.56 MHz ccrf discharges in nitrogen, produced within a cylindrical, parallel-plate GEC-type reactor, for a wide range of gas pressures and applied rf voltages. We use a two-dimensional time-dependent fluid-type code to describe the transport of electrons and positive ions N_2^+ and N_4^+, in the reactor under study. The charged particle transport model is coupled to a very complete zero-dimensional kinetic code for nitrogen gas, which solves the electron Boltzmann equation and the rate balance equations of 45 vibrationaly excited states and 7 electronically excited states of the nitrogen molecule. Simulations yield the self-consistent dc-bias potential, the effective power coupled to the plasma, and the two-dimensional spatial distributions for (i) the densities and fluxes with the charged particles and the electron mean energy, (ii) the densities of the most relevant molecular states of nitrogen, and (iii) the rf plasma potential. Results reveal a strong coupling between discharge and kinetic features, as electrons are mainly produced via associative ionization mechanisms involving the molecular metastable states $N_2(A)$ and $N_2(a')$. Model results are compared with experimental measurements of the mean electron density and the self-bias potential at the polarized electrode.

Keywords: Plasma, Nitrogen, Radio-frequency discharges

The dependence of the target erosion depth on the direction of magnetic induction in a planar magnetron discharge

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Abstract

The target erosion depth, h(r), is measured along a radius of a circular magnetron target and the magnetic induction vector is mapped.

A simplified model of the discharge is developed in the present work. The model shows that the erosion depth along a target radius is well defined by a relation of the type, h(r)=hm[1-exp(-bcotg θ(r,L)], where hm is the maximum target erosion depth, b is a characteristic parameter of the discharge, L is the thickness of the cathode sheath and θ the angle made by the magnetic induction and the target surface.

The relation obtained for the dependence of the target erosion depth on the direction of the magnetic induction fits well the experimental results.

Keywords: target erosion depth; planar magnetron discharge.

Optical Properties of Acetylene-Based Diamond-like-Carbon Films

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Abstract

Diamond-like carbon (DLC) films can be deposited at higher deposition rates when acetylene C_2H_2 is used instead of the commonly employed CH_4 source gas. However, film quality may suffer as far as optical absorption and transport properties are concerned.

We have decomposed a mixture of acetylene and hydrogen by conventional plasma CVD at 13.56 MHz at low substrate temperature up to 400 ºC. Indeed, with acetylene alone the deposition rate reached values as high as 80 nm per minute. Films from pure acetylene showed a yellowish colour consistent with appreciable optical absorption in the 400 to 700 nm range. Broad Urbach tails were obtained from the absorption spectra. For example, with hydrogen dilution at 98 % the deposition rate dropped by a factor of 10, on the other hand the transparency improved substantially. On selected films we measured the photo and dark conductivity.

Films were further analysed by infrared absorption spectra between 400 and 4500 cm^{-1}. To estimate the relative amount of graphitic material the individual contributions of sp^3-CH, sp^3-$CH_{2,3}$ and sp^2 species were determined from the individual peak intensities at 2850, 2920, and 2950 cm^{-1}, respectively. We are investigating further improvements of film quality by increasing plasma power and hydrogen dilution to reduce remaining graphitic components.

Acknowledgements: This work was supported by Fundação para a Ciência e a Tecnologia (FCT) through project POCTI/CTM/41317/2001 (SICAL).

Keywords: Amorphous carbon, DLC, Acetylene, Plasma-enhanced chemical vapour deposition

Atomic Ionization Cross Sections by Electron Impact

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Abstract

There is a need for reliable theoretical methods to calculate electron-impact total ionization cross sections for a large number of neutral atoms and ions with open and closed shell structures. These cross sections are used in a wide range of scientific and industrial applications, such as astrophysical plasmas, atmospheric science, X-ray lasers, magnetic fusion, radiation physics, semiconductor fabrication, accelerator physics and tumour therapy physics.

The binary-encounter-Bethe (BEB) model [1], using an analytical formula that requires only two atomic constants, the binding energy and kinetic energy of the electrons, generates direct ionization cross sections for any neutral atom (or molecule), which are reliable in intensity (±15%) and shape from the ionization threshold to a few keV in the incident energy [2], or to thousands keV if we consider its relativistic version (RBEB) [3].

In this work, the BEB/RBEB model was used to calculate cross sections for L-shell ionization of Xe and Kr by electron impact. Precise binding and kinetic energies of electrons in the L-subshells were obtained with the Multi Configuration Dirac-Fock (MCDF) method without the inclusion of correlation orbitals.

Keywords: cross sections, electron impact, L-Shell

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Singularities in the I-V Characteristics in a DC Magnetron Discharge

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Abstract.

The DC abnormal glow discharges with magnetic field assistance are particularly useful in magnetron sputtering deposition techniques. A magnetron cathode, based on permanent NdFeB magnets, has been constructed to allow the control of the magnetic configuration. A confinement power parameter CB [1], provides a quantitative quality factor for the magnetic trapping near to the cathode.

Sets of current − voltage, I-V curves, where measured for Argon glow discharges on Copper at several confinement power values and several pressures.

In this work and for the first time, singularities or steps where observed in the current−voltage I-V, characteristics taken at constant pressure values and at several confinement power values. The occurrence and sharpness of the steps in the I-V curves depend on the magnetic field confinement and the discharge pressure. A quantitative model is proposed relating the observed steps with the threshold behaviour of sputtering of the target atoms by fast particles from the cold plasma. It takes into account cathode sheath thickness dependencies on voltage and pressure and it explains quantitatively the variation of the voltage step occurrence with the pressure and is consistent with the threshold energy of 27 eV [2] proposed in the literature for Cu low energy sputtering by Argon particles at perpendicular incidence.

Two plasma regimes are proposed for this discharge, before and after the step. At voltages before the step the plasma is sustained by grazing incident Argon ions with low energies and low sputtering rates result. After the step incident Argon ions with energies higher than the sputtering threshold at normal incidence produce higher sputtering rates and increase the eficiency of the coating process.

Keywords: abnormal glow discharge; magnetic confinement; sputtering

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Modification of alumina porous support for catalytic proposes

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Abstract

Commercial γ-alumina porous supports with average nominal pore size of 0.02 μm (Anodisc 13 from Whatman) have been modified by impregnation with two different percentages of platinum (2% and 20%) and calcinated (573 K and 873 K) for possible application in catalytic reforming of propane.

The unmodified and modified alumina supports were structurally characterized in order to determine the presence of Pt into the pores and, consequently, its possible application as a nanoreactor. Structural and physic-chemical characterizations were carried out by SEM and TEM micrographs, X-Ray Photoelectron Spectroscopy (XPS) and Infrared Spectroscopy of Fourier Transformate (FTIR).

A slight reduction in surface porosity as a result of platinum impregnation and calcination (2 % Pt and 873 K) was determined from SEM micrographs, while Pt nanoparticles into the pores can also be observed in TEM micrographs; “in situ” (transport) measurements were also carried out to corroborate porosity reduction [1]. XPS analysis allows the determination of the atomic concentrations of the elements present on the surface of the studied samples; for the Pt modified supports a peak at a B.E. of 315 eV (Pt (II)) and a shoulder at 317 eV (Pt (IV)) were obtained. CO-interactions with Pt-reduced centres (Pt ^{o}) were not detected from CO-FTIR spectra. The highest Pt-loading favours the H_2 formation, above 873 K, from C_3H_8-CO_2-reforming.

Acknowledgements: To CICYT (Project MAT2007-65065) and Junta de Andalucía (Excellence Project P06-FQM-1764) for partial financial support.

Keywords: XPS, FTIR, TEM, catalyst.

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Control of the substrate bias in dc PVD processes using insulator substrates

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Abstract

It is well known that ion bombardment during the growth of thin films has a strong influence on the properties of thin films, such as morphology, composition, structure, stress, electrical conductivity, and others. Therefore, an accurate control of substrate bias is needed in order to deposit films with the desired properties. For insulator substrates, dc biasing the substrate holder is useless, since the surface shall not follow the applied bias but it will be at the non-controlled floating potential.

In this work we present a method for the effective control of the substrate bias in dc PVD processes with insulator substrates based on placing a metallic grid at a certain distance from the non-conductive surfaces to be coated. The desire negative bias is applied to this metallic grid which accelerates ions from the plasma and directs them to the surface to cover. This method has been applied successfully to the deposition of TiN coatings on glass and decorative ceramics. The influence of applying different negative bias values on coatings stress and adhesion as well as the shadowing effects of the grid are discussed.

Keywords: PVD, insulator substrates, bias, adhesion.

Structure and morphology of TiB_2 duplex coatings deposited over X40CrMoV 5-1-1 steel by DC magnetron sputtering

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Abstract

The deposition of titanium diboride (TiB_2) films over tool steel substrate (AISI H13 premium/EN X40 CrMoV 5-1-1) is being investigated due to its excellent corrosion resistance and chemical stability against liquid aluminium. The use of nitrided steels as substrates for TiB2 deposition may contribute to increase its adhesion and the overall steel resistance in applications such as forging, extrusion and die casting of aluminium.

The aim of the present work is the production and characterization of duplex coatings and their soldering resistance assessment in the contact with liquid aluminium. Disc shaped hot work tool steel substrates were quenched and tempered (QT) in vacuum furnaces before nitriding (QTN). Quenching was done with a forced flux of nitrogen at 3 bar over-pressure from 1050°C. Nitriding was performed at 540°C using the Allnit® low pressure technology. The substrates were characterized using Rockwell C and Vickers hardness indentation tests and also by means of metallographic and X-Ray diffraction analysis.

The duplex coatings were obtained by the PVD deposition of TiB_2 films over the heat treated and nitrided steel (QTN) using non-reactive DC magnetron sputtering from a TiB_2 target. The studied deposition parameters were the target/substrate distance, substrate temperature, the emission current and the substrate bias voltage. Selected films were tested for resistance to liquid aluminium soldering by immersion tests, and compared with standard QT and QTN steels. The coatings were characterized by X-Ray diffraction and scanning electron microscopy with EDS analysis, before and after immersion testing.

Well structured and crystalline TiB_2 films were obtained for selected deposition conditions. Best results were obtained with positive bias voltage at the highest emission current used (0.5 A). Substrate heating always results in increased crystallinity and homogeneity of the films. The best duplex coatings tested in the contact with liquid aluminium proved to have increased soldering resistance relatively to the uncoated QT and QTN steels.

Keywords: TiB_2, Duplex coatings, DC-Sputtering

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Comparative testing of amorphous carbon layers for bio-tribological aplications

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Abstract

Amorphous hydrogenated carbon (a-C:H), also known as diamond-like carbon (DLC), is a class of materials with excellent mechanical, tribological and biological properties. Friction coefficient values between steel and DLC coatings are very low 0,05. [1] Due to its bio- and haemocompatible nature there is a growing interest in the application of DLC on orthopedic and other implants.

This contribution is oriented to tribological behaviour of hard DLC coatings made by physical vapour deposition on CrMoCo alloy-substrate [2]. The study is oriented to evaluation of DLC coatings in the dependence on sample shape. Coatings were deposited by PVD method on CrMoCo alloy-substrate system. The aim of this contribution is to compare the tribological properties for flat and shaped engineering surfaces with DLC layers – Fig. 1.

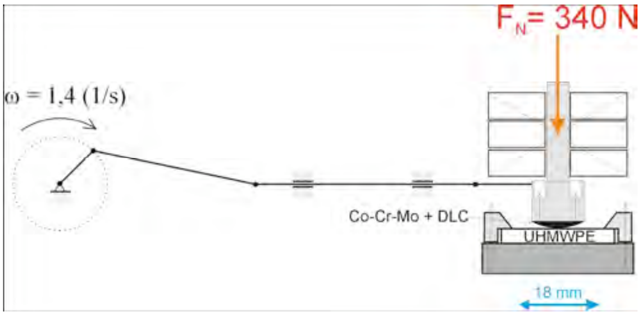

Fig. 1 Scheme of test

The objective is to contribute to the design of surface engineering systems with optimised friction and minimized wear with longer lifetime or in the new applications.

Keywords: (DLC, mechanical and tribological properties)

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STM study of the TiO_2(110)-(1x2) surface reconstruction

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Abstract

The study of the metal oxide surface is of a great scientific interest due to their huge technological applications. In particular, the rutile TiO_2 has been the subject of numerous investigations since it is relevant for a wide variety of fields, ranging from catalysis to fundamental chemistry. Amongst the different surfaces of the single-crystalline TiO_2 system, the (110) face is the most stable one being its 1x1 structure extensively studied in the last decades. This well-known (1x1) structure transforms into a long ordered reconstruction with a (1x2) symmetry after reducing the TiO_2(110) surface by annealing up to about 800ºC. The understanding of the basic properties of the non-stoichiometric (1x2) TiO_2(110) reconstruction is still nowadays unclear. Recently, the atomic configuration of the 1x2 TiO_2(110) surface has been elucidated by combining scanning tunneling microscopy (STM), quantitative LEED and density functional theory (DFT) [1]. The resulting model consists of added Ti_2O_3 rows along the [001] direction in agreement with a previous proposal by Onishi et al [2].

In the present work, the non-stoichiometric (1x2) TiO_2(110) reconstruction was studied by STM and scanning tunneling spectroscopy (STS). The recorded images show monoatomic steps, wide terraces and no cross-link features as it can be observed in Figure 1. STS current-voltage curves were recorded to obtain information on the local density of states (DOS).

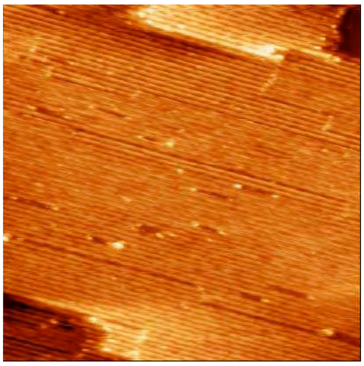

Figure 1: STM image of the 1x2 TiO_2 (110) surface reconstruction

Keywords: Scanning Tunnelling microscopy, titanium oxide

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Repercussion of the surface characteristics of an activated carbon fibers in the adsorption of hydrogen.

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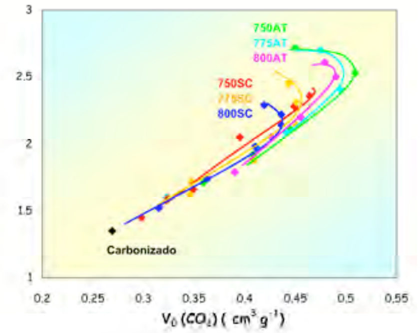
Abstract

In recent years, interest in the use of hydrogen as an alternative to fossil fuels has increased greatly. The main reasons for this are the foreseeable depletion of the world's reserves of crude oil and the need to reduce the levels of pollutants responsible for the greenhouse effect.

However, its widespread deployment is dependent upon the development of a safe, simple and feasible storage method. According to the US Department of Energy (DOE), the goal sought is 6 and 9 wt% storage in 2010 and 2015 respectively [1].

In light of the above, one of the options that has kindled the greatest interest is the storage of hydrogen as a gas in porous solids. Different materials have been investigated, such as activated carbon, activated carbon fibbers and nanostructured materials, activated carbon fibres being the materials that seem to be most promising for the future.

In the present work, carbon fibbers were physically activated with CO_2 in the supercritical state at atmospheric pressure at different temperatures -750, 775 and 800ºC- until different degrees of activation were achieved. The results reflect the importance of the textural characteristics of the activated carbon fibbers on the hydrogen storage capacity.



The figure shows the percentage of H_2 stored by the different series of fibbers activated with supercritical CO_2 at atmospheric pressure as a function of the micropore volume (V0(CO_2).

Our results suggest that there is a close relationship between the surface characteristics -the BET surface area and micropore volume- and the hydrogen storage capacity. They also suggest that there is an optimum micropore volume value with which it would be possible to achieve maximum storage capacity.

[1] U.S.DOE Energy Efficiency and Renewable Energy (EERE)
(http://www.eere.energy.gov/hydrogen)

Acknowledgements

The authors thanks Ministerio de Ciencia y Tecnología , proyect CTQ2006-00759/PPQ and Junta de Castilla y León, proyect SA054A07, for financial support

Keywords: Hydrogen storage, activated carbon fibbers.

Study of the surface characteristics of a carbon fibber activated with supercritical water.

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Abstract

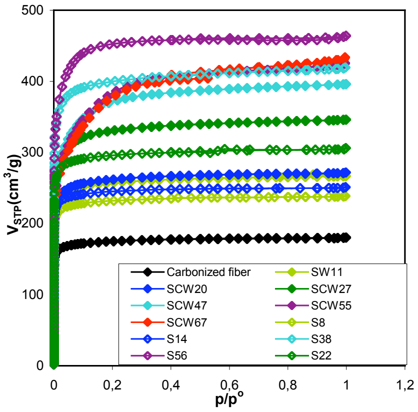
Activated carbon fibbers (ACFs) are materials that have aroused considerable attention owing to their properties as adsorbents. These materials exhibit a series of advantages over other carbonaceous adsorbent materials, such as a high surface area, a narrow pore size distribution, and pores with small diameters. These properties afford such fibbers high capacities and rates of adsorption.

The most usual method used to prepare ACFs is physical activation with steam or CO_2.

Here we report a comparative study of the textural characteristics of fibbers activated with supercritical water (SCW) at 670- 710ºC and 240-250 bar and with steam. These textural characteristics were determined from analysis of the N_2 and CO_2 adsorption isotherms at 77 K and 273 K respectively and by means of scanning electron microscopy (SEM).

The figure shows the N_2 adsorption-desorption isotherms at 77 K for samples with different degrees of activation with SCW and with steam at 710 ºC.

The results indicate that with both procedures it is possible to obtain highly microporous ACFs, with a large surface area and a high micropore volume. The fibbers activated with SCW show a broader microporosity than those activated with steam. This reveals that activation with SCW leads to an increase in porosity [1].



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Acknowledgements

The authors thanks Ministerio de Ciencia y Tecnología , proyect CTQ2006-00759/PPQ and Junta de Castilla y León, proyect SA054A07, for financial support.

Keywords: activated carbon fibbers and supercritical water.

XPS and ATR/MIR studies of functionalized cellulose films

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Abstract

Surface studies deal a lot with vacuum technology since surface analysis techniques frequently demand high or even ultra-high vacuum (UHV) conditions. However, not all molecular modifications can be generated under UHV. Actually, surface modifications towards functionalization are normally performed in liquid medium and under atmospheric conditions. Fourier Transform Infrared Spectroscopy (FTIRS) is then used as a current method of analysis. In the specific case of semiconductor substrates, mostly transparent in the mid-infrared region, Attenuated Total Reflection in Multiple Internal Reflections (ATR/MIR) is the appropriate mode of FTIRS to probe reactions occurring on the surface [1].

Lately, ultrathin films of cellulose could be produced by spin-coating on semiconductor surfaces, namely on GaAs (100). The films were chemically modified and the kinetics of several reactions, well-known in the chemistry of massive cellulose, could be followed in real time by ATR/MIR. Furthermore, crossed information obtained from X-ray photoelectron spectroscopy (XPS) with ATR/MIR data revealed to be particularly useful [2]. This method is applied in the present study where the surface of cellulose films was modified with grafting metalloporphyrins.

Actually, metalloporphyrins are very interesting molecular entities for chemical sensors, as they contain the specific ability to coordinate different gas molecules as CO_2 or NO [3]. The protoporphyrin chosen is the hemin, a natural protoporphyrin (IX), which contains an iron atom (FePP) in the centre of its conjugated crown. The reactivity of the cellulose surface towards the hemin was enhanced by making the hemin react with N,N'-carbonyldiimidazole (CDI). This enabled the grafting of the porphyrin through the alcohol groups of the cellulose chain. The effect of different spacers between the porphyrin and the cellulose was also investigated. The highest level of FePP chemical adsorption was achieved when both cellulose surface and FePP were previously activated by CDI and 1,8-diaminooctane (DAO) used as spacer [4].

Keywords: Cellulose; Functionalization; FePP; XPS; ATR/MIR

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Reversible Phase Transition:Indium Chains on Silicon

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Abstract

The In/Si(111)-(4x1) surface is a paradigmatic example of a quasi-one-dimensional system showing a reversible structural and electronic (metal-insulator) phase transition when the temperature is lowered. We have used first-principles simulation techniques [1][2] to uncover the atomic and electronic origin of this controversial transition. Our calculations show that the ground state consists of insulating (4x2) indium chains with a weak interchain coupling that induces opposite shear distortions in alternate chains [3]. Our earlier first-principles molecular dynamics simulations show that the (4x1)↔(8x2) phase transition is due to the "dynamical fluctuations" the system undergoes when, at high temperature, it fluctuates chaotically between degenerate ground states [4]. The metallicity of the In/Si(111)-(4x1) surface is related to the low energy cost for the shear distortion. Now, the combination of new experimental and theoretical STM-STS results confirms the validity of our "dynamical fluctuations" model.

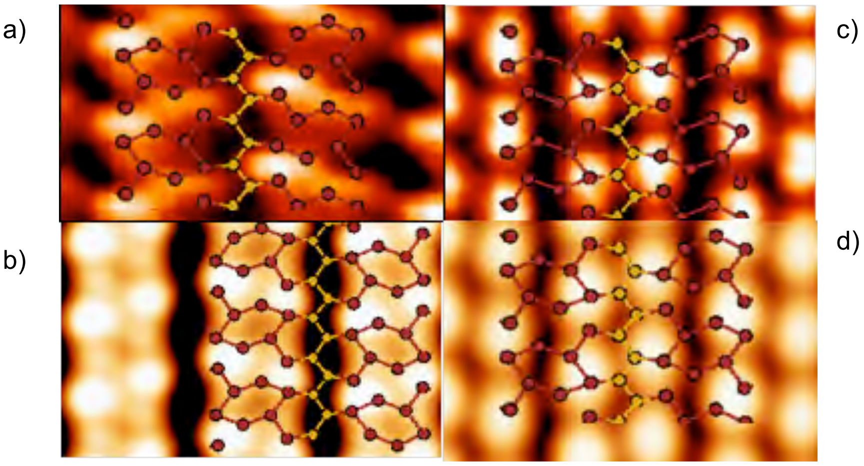

Figure: Experimental (a,c) and theoretical (b,d) STM images, for -0.5V (a,b) and 1.5 V (c,d)

Keywords: STM, first-principles, phase transition

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Interface barrier formation for C60 or benzene adsorbed on Au(111): the unified IDIS-model and the role of the "pillow" dipole.

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Abstract

The adsorption of a C60 (or a benzene) monolayer on Au(111) is analyzed using a first-principles DFT-approach based on a local-orbital Fireball technique [1]. In both cases, adsorption energies, barrier heights, Fermi levels and Charge Neutrality Levels are calculated and compared with the unified IDIS (Induced Density of Interface States)-model [2-4]. In particular, induced interface dipoles associated with the charge transfer and the "pillow" effect are calculated and shown to depend crucially on the interface screening parameter, S. We show that C60 and benzene belong to two extreme cases; while in C60 the surface screening is large, in benzene it is small. This discriminates about the role played by the "pillow"-dipole in the formation of the barrier height: we show that this dipole plays an important role in benzene [5] but its effect is minor in C60. In this last case, we show that the Fermi level is close to the molecule Charge Neutrality Level.

Keywords: Organic interfaces, C60, benzene, Charge neutrality level

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First Principles Study of Point Defects in Titanium Oxycarbides

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Abstract

Transition metal oxycarbides are known as refractory metal compounds. They combine metallic properties like conductivity with typical properties of covalent bonded materials such as extreme hardness. Changing the O/C ratio it is possible to obtain a combination unique of properties, which attracted the attention to these materials for a wide range of high technological applications. Due to the methods used in the growth of these materials they present a high concentration of defects, especially vacancies, which play an important role in the material properties.

In this work we used first principles density functional theory to study the energetic of point defects in the titanium oxycarbides. We present calculations of the formation energies of Ti, C and O vacancies in TiC_xO_{1-x} compounds. We also investigated formation energies of C and O substitutional impurities. The calculations reveal that C, O vacancies are likely to occur in Ti-rich conditions, while Ti vacancies in O-rich conditions.

Keywords: Oxycarbide, Defects, Density Functional Theory

Properties of indium tin oxide thin films deposited on acrylics substrates by Ion beam assisted deposition technique

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Abstract

Indium tin oxide (ITO) thin films have been deposited onto acrylics (PMMA) substrates by ion beam assisted deposition technique at different oxygen flows. The structural, optical and electrical properties of the deposited films have been characterized by X-ray diffraction, transmittance, FTIR, ellipometry and Hall effect measurements. The optical constants of the deposited films have been calculated by fitting the ellipsometric spectra. The effects of the oxygen flow on the properties of the deposited films have been studied. The oxygen flow is varied from 20 to 50 sccm. All the films show amorphous structure. The film prepared at high oxygen flow gives high optical transmittance, high refractive index and low extinction coefficient. Although the maximum transmittance (91% at 500 nm) is obtained for ITO film deposited at 50 sccm oxygen flow, it gives a high electrical resistivity (15,8 x 10-3 Ω-cm). By considering both the transmittance and the electrical resistivity, It has been found that 40 sccm oxygen flow is the optimum value for depositing ITO films. At this condition, the ITO films with 87% transmittance at 500 nm and 2,8 x 10^{-3} Ω-cm electrical resistivity have been obtained. It shows that ion beam assisted deposition technique is a suitable technique to deposit high quality ITO films at low substrate temperature.

Key Words: Thin Film; PMMA; Ion beam assisted deposition; Indium tin Oxide.

An Investigation of the Nano-structure and Electrical Properties of Annealed Ti-oxide Thin Films as a Function of Film Thickness

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Abstract

Ti films of different thickness ranging from 12.3 to 246.2 nm were deposited, using resistive heat method and post-annealed at 573 K temperature with a flow of oxygen. The nano-structures of the films were obtained using X-ray diffraction (XRD) and atomic force microscopy (AFM). The Rutherford Back scattering technique was used for the determination of the films' thicknesses. The results showed that the grain sizes, the films' surface roughness increase by annealing procedure. The XRD patterns showed an increase of the (004) orientation of the anatase phase of TiO_2 with thickness. The resistivity, Hall constant, and the mobility of the carriers decreased and carriers concentration increased with increasing the film thickness, while carriers concentration and mobility of the carriers decrease and the Hall constant and resistivity increase by annealing the films.

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Microstructural features of MgF_2, ZnS thin films and MgF_2/ZnS multilayers

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Abstract

The microstructure of MgF_2 and ZnS thin films, as well as, MgF_2/ZnS multilayers for optical coating, has been studied. All samples have been obtained by thermal evaporation and submitted to a post deposition thermal annealing treatment between 200 and 400 ºC. Grain size and microstrain haven been calculated from X-ray diffraction by applying different fitting equations (Scherrer, Scherrer-Wilson and Williamson-Hall). The X ray diffraction spectra have been obtained at grazing incidence. Depending on the fitting expression, different quantitative results in grain size and microstrains have been obtained but the same behaviour with the temperature is observed.

Thickness dependence evolutional process has been carried on by measuring a set of diffraction pattern under varying incident angles in the range 0.5 to 2º. Behaviour of the structure with the thickness has been observed by applying different concepts to calculate the penetration depth (1).

Finally, transversal images have been observed by means of SEM by exposition of transversal cuts of thin films and multilayers respectively. Columnar grains are observed along the thickness in all the samples. The evolution of the grain size and microstrain with thermal annealing at the different temperatures are presented. Differences are observed in the structure of the materials in single thin films and when they are part of multilayers. The interface strains in the multilayers case is analyzed in function to the layers number of the different samples.

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Keywords: thin films, multilayers, microstructure, grain size

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Structural characterization of lead niobate-cobalt ferrite nanogranular composites deposited by pulsed laser ablation

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Abstract

Multiferroic materials presenting a coupling between the electric and magnetic degrees of freedom have been attracting much scientific and technological interest. By combining a piezoelectric ceramic and a magnetostrictive material the elastic interactions between the phases provide the coupling mechanism inducing a magnetoelectric behavior. Here, nanocomposites of cobalt ferrite ($CoFe_2O_4$-magnetostrictive) dispersed in a lead niobate ($PbNb_2O_6$-piezoelectric) matrix were prepared and their structure was characterized. The films were deposited by pulsed laser ablation, with a KrF excimer laser (wavelength λ = 248 nm), for different cobalt ferrite concentrations (from 20% to 80% CoFe2O4). The oxygen pressure during deposition was 1 mbar and the substrate temperature was 600ºC. Their structure was studied by X-ray diffraction.

The films are polycrystalline and composed by a mixture of lead niobate with the orthorhombic structure and cobalt ferrite with the cubic inverse spinel structure. The grain sizes measured from the X-ray diffraction peak widths for both phases are in the range 25 nm – 30 nm for $CoFe_2O_4$ and 60 nm – 90 nm for $PbNb_2O_6$. The lattice parameters of the lead niobate phase do not change much for cobalt ferrite concentrations up to 60%, being near from the bulk values (a = 17.03 Å, b = 17.93 Å, c = 3.868 Å). On the other hand, the lattice parameter of the $CoFe_2O_4$ phase, obtained from the (511) peak position, was ~ 8.23 Å – 8.26 Å in the cobalt ferrite concentration region 20%-60%. Comparing with the bulk cubic inverse spinnel $CoFe_2O_4$ lattice parameter (a=8.3874Å), it is observed that in the films the cobalt ferrite is under compressive strain. For higher $CoFe_2O_4$ concentrations this strain relaxes and the lattice parameter is near the bulk value. A detailed characterization of the nanocomposites structure is presented.

Keywords: Nanogranular composites, piezoelectric-magnetostrictive, laser ablation, structure

Structural characterisation of multifunctional biocompatible Ti-Si-C-ON coatings

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Abstract

Materials to be used as load-bearing implants must possess enhanced physical, chemical, mechanical, tribological and biological properties. One alternative to obtain improved implants is the production of multifunctional coatings, were different elements and compounds can be synergetically combined [1]. Ti-based coatings are widely used in biomedical applications due to the high hardness and excellent wear and corrosion resistance (TiN) or the outstanding biocompatibility (TiO_2). Furthermore, the addition of silicon to the TiN structure can lead to a major improvement of the hardness, and carbon is a well established material in biomedical applications because in amorphous state can strongly reduce the friction coefficient of the material while keeping a good biocompatibility. Finally, recent works have shown that Ti-Si-C based coatings are promising systems due to their excellent mechanical and tribological performance [2].

In this work we study multicomponent Ti-Si-C-ON coatings deposited by DC reactive magnetron sputtering. Different partial pressures of oxygen and nitrogen and changes in the applied power of TiSi and TiC cathodes lead to a wide range of coating composition and structures. The stoichiometry of the films was investigated by means of Rutherford Backscattering Spectrometry (RBS) and Glow Discharge Optical Emission Spectroscopy (GDOES), while X-ray diffraction (θ-2θ and Glancing incidence) was applied to investigate their structure. Chemical bonding information was obtained by Raman and X-ray Photoelectron Spectroscopy (XPS). The characterisation of the deposited films was completed by a surface topography and morphology analysis performed by Atomic Force Microscopy (AFM). We will discuss the correlation of the structural properties of the coatings to their potential biomedical applications, as for example inhibitors of biofilm formation.

Keywords: Multifunctional coatings, structural characterisation

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Influence of the target composition on reactively sputtered titanium oxide films

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Abstract

Titanium dioxide (TiO_2) thin films have many interesting properties and are used in various applications. High refractive index of TiO_2 makes it attractive for the glass coating industry, where it is used in low-emissivity and antireflective coatings. Magnetron sputtering is the most common deposition technique for large area coatings. Therefore, high deposition rate processes are of obvious interest.

It has been shown previously that high rate can be achieved using substoichiometric targets TiO_x. This work deals with reactive magnetron sputtering of titanium oxide films from targets of different composition. We report on the film properties of films deposited from targets of different composition.

The DC deposition process with targets of different oxygen content has been investigated. Films were prepared at various oxygen flows. All films were deposited onto glass and silicon substrates with no external heating. The elemental compositions and structures of films were evaluated by means of X-ray photoelectron spectroscopy, Elastic recoil detection analysis and X-ray diffraction. The optical properties of the films in visible range were investigated. The influence of the target composition and sputtering condition on film properties is discussed. Model of the reactive deposition process with substoichiometric targets has been developed. The computer simulations are compared with experimental results.

Keywords: Magnetron sputtering, titanium dioxide, high rate deposition

Smart Thermochromic Coatings for Energy Efficient Windows

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Abstract

The latest approach on the improvement the energy efficiency of buildings is based on the use of thermochromic coatings on so-called “smart” windows. These coatings possess the ability of changing their optical properties as a consequence of a reversible structural transformation when going through a critical temperature. Vanadium dioxide is an example of a transparent thermochromic material which is a promising candidate for this kind of application. The change on its optical and also electrical properties takes place at 68ºC as a result of a first-order structural transition, known as Mott transition [1], going from a monoclinic to a tetragonal phase on heating. The low temperature semiconducting phase which is transparent to radiation in the visible and infrared wavelength range maximizes the heating due to blackbody radiation, while the metallic high temperature phase blocks the infrared radiation and maintains at the same time the transparency required, in the visible range, to keep an environment of natural light. A transition temperature of 68ºC is too high for this application and must therefore be reduced. Tungsten-doping of VO_2 has demonstrated to decrease the transition temperature in the greatest extent, when compared with other metals, and has therefore been the focus of most of the research [2].

In this study, we have prepared VO_2 thin films onto SiO_2-coated float-glass substrates by reactive direct current (DC) magnetron sputtering. Different processing conditions have been chosen in order to obtain thin films with different structural and consequently different optical properties. Pure VO_2 thin films and also doped with different W at.% and consequent dissimilar switching temperatures, were successfully deposited. The films were characterized in terms of crystal structure and texture by x-ray diffraction (XRD) and the morphology of the surface has been analyzed and quantified by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The optical/thermochromic behavior of the different films has been studied by optical spectrophotometry in the UV-VIS-NIR range.

Keywords: Thermochromic films; Vanadium Dioxide; Magnetron sputtering; Smart windows

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Properties of the Amorphous Carbon Films Sputtered on Steel Substrates

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Abstract

Amorphous carbon films were deposited in pure argon on chromium steel substrates in the vacuum plant Leybold by DC magnetron sputtering from pure carbon target with glued either Ti or Si blades. The C films of thickness about 1.5 – 2.5 μm contained either Ti or Si, added during sputtering at different deposition conditions: with a floating potential of substrate or with ion bombarding by a negative bias potential from RF source connected to the substrate holder. Mechanical properties such as hardness, elastic modulus, and elastic work at maximal load 10 mN were measured on the Fischerscope H100 equipped with Vicker's diamond indenter. Scratch or adhesion indentation tests were performed with Rockwell diamond tip on the CSEM REVETEST apparatus at increasing normal load up to 80 N in scratching and at loading power 20, 50, and 150 N for indentations. The scratch tests were performed only for a-C:Ti films. Coating wear resistance and coefficient of friction were examined by pin-on-disc tests. Mechanical properties showed dependency on deposition conditions and composition of the films. Ion bombardment during film growth is the principal factor which influences mechanical properties of amorphous carbon coatings.

Keywords: amorphous carbon films, mechanical and tribological properties, composition dependency

Structural and Transport Modifications of Cellulosic Membranes Due to Gamma-Radiation

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Abstract

Cellulose and its derivatives are common materials for membrane manufacture [1]; particularly, regenerated cellulose (RC) is the base of many dense membranes used for dialysis /haemodialysis processes, since due to high hydrophilic character it allows the transport of ions and small particles but reject macromolecules. For these applications, temperature and radiation (ultra-violet and gamma-radiation) are commonly used in connection to membrane cleaning/sterilization purposes, but those protocols can cause changes in the structure and electrical parameters of the membrane matrix, which might affect the transport of electrolytes solutions.

This work studies structural and transport modifications of three RC dense membranes as a result of γ-irradiation. The membranes have different cellulose content (0.03 kg/m^2 (RC-3), 0.05 kg/m^2 (RC-5) and 0.06 kg/m^2 (RC-6)), geometrical (wet thickness and swelling degree) and transport (hydraulic and diffusive permeabilities) parameters. Membrane irradiation was performed by using a therapeutic 60Co Cobalt Unit (average photon energy of 1.27 MeV) with an irradiation doses of 80 J/kg (RC-X-Ir80 samples). Structural characterization was carried out by Infrared Spectroscopy of Fourier Transformate (FTIR), X-ray diffraction and X-ray photoelectron spectroscopy (XPS), while changes in diffusional permeability (Ps) determined from NaCl diffusion measurements at different feed concentrations were considered in order to estimate the effect of radiation on solute transport. A reduction of Ps values for the irradiated membranes was obtained, which seems indicate a compaction of the cellulose chains. Slight changes in FTIR spectra and diffraction peaks when untreated and irradiated samples are compare also indicate membranes modification, while XPS analysis indicates the surface cleaning as main result of irradiation.

Acknowledgements: To CICYT (Project MAT2007-65065) and Junta de Andalucía (Excellence Project P06-FQM-1764) for financial support.

Keywords: FTIR, X-ray diffraction, permeability, gamma-irradiation.

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Morphology Characterization of (Al, Ti) and [(Al, Ti) N] thin films with Gradient Depth Profile Composition.

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Abstract

Thin films of (Al,Ti,N) produced with DC reactive magnetron sputtering can significantly improve electrochemical and biocompatibility properties of the base metal alloy[1,2,3].

The composition depth gradient of Al and Ti in the (Al,Ti,N) films may induce accrued wear resistance, adhesion and chemical inertia. These films were obtained by co-deposition from two DC magnetron cathodes, with independent plasma power supplies, controlled by a custom made piece of software, which allows real-time, simultaneous and independent control of the deposition rates from both cathodes [4].

In this work the morphology of (Al,Ti) and (Al,Ti,N) films, with thickness in the range of 700 to 900nm and gradient depth profile composition, were analysed by AFM (atomic force microscopy). These composition depth gradient films in starts either with pure Al or pure Ti were deposited on Si and glass at room temperature without bias. To obtain (Al,Ti,N) thin films with gradient composition depth gradient, Ar, N_2 mixture (25% nitrogen partial pressure) was used in the discharge.

For the same binary alloy composition, the evolution of topographic parameters like RMS roughness and average height shows remarkably different behaviour, depending on the substrate were the film was grown, the top metal layer (interface film-air) and N_2 addition in the plasma.

Keywords: AFM, Thin film, Topography, Al-Ti alloy

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Development of sputtered Shape Memory Alloy (SMA) Ni-Ti films for actuation in ice cooled environments

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Abstract

Due to the high sensitivity of Ni-Ti films to environmental changes, e.g. thermal, and/or to stress, they are ideal materials for applications in microsensors.

Widening the scope of previous experiments concerning the influence of the type of substrate (single-crystal Si, polycrystalline Si) on the structural development of the Ni-Ti films during crystallization, here the incorporation of a SiNx intermediate layer was tested. Like in the previous work, the crystallization behaviour (at a constant temperature of 430°C) has been studied by X-ray diffraction in grazing incidence geometry off-plane (GIXD) at a synchrotron-radiation beamline. The GIXD patterns obtained during the annealing process of the Ni-Ti polycrystalline films revealed mainly an austenitic structure (B2 phase) and the precipitation of Ni_4Ti_3. The results have also shown that the presence of an intermediate layer of SiN_x enhances the crystallization process of the Ni-Ti sputtered films when compared with the films deposited directly on single-crystal Si (with native oxide).

The phase transformation behaviour of the Ni-Ti film on SiNx has been evaluated by XRD in off-plane Bragg-Brentano geometry during cooling (RT → -40°C) and heating (-40°C → RT). It has been observed that a big fraction of the Ni-Ti film is already transformed to R-phase at 9°C (austenitic at RT) as well as a very small temperature hysteresis for the B2 ⇔ R-phase transformation.

After the characterization described earlier, the film was removed from the substrate and showed free standing a pronounced “two-way” shape memory effect (SME). In the austenitic state the film presents a flat shape. During cooling, by reducing its distance from ice cubes, starts bending exhibiting a final curled shape (yet without touching the ice). On heating it recovers its flat shape. We attribute the nature of this “two-way” SME to the Ni_4Ti_3 precipitates that formed during the heat treatment.

The influence of e-beam irradiation on characteristics of MIS structure with HfSiO:N-HfTiO:N high-k laminate dielectric stack

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Abstract

We study E-beam irradiation effects in the dose range of 1-8000μC/cm^2 on Metal-Insulator-Semiconductor (MIS) structures with 3 and 4.8 nm dielectric stacks based on double layer high-k materials HfSiO:N and HfTiO:N. The dielectric layers were deposited on p-Si by electron beam evaporation. The layers remain amorphous after annealing at 750 °C in oxygen area for 1 min. Au and Ti/Au are used as gate and back electrodes, respectively. E-beam irradiation is found to initiate several effects: i) introduce positive traps in the dielectric leading to the shift in capacitance-voltage (C-V) curves to the negative applied bias and increase of hysteresis independently on insulator stack thickness; ii) The leakage current density (JL) does not experience any type of influence in the structure with thin 3 nm thick insulator stack. The thick (4.8 nm) dielectric stack exhibits two additional effects: a) a slight decrease in the effective dielectric constant and b) large reduction in the JL with irradiation. Both effects are due to possible formation of the low dielectric constant SiOx type interfacial layer. Low temperature annealing (at 300 °C in air for 2 min) turns back the C-V characteristics and therefore almost recovers the pre-irradiated parameters, while it does not influence on the leakage current density. Based on the analysis of J-V (or α-V, where α is defined as $\alpha = d[\ln(J)] / d[\ln(V)]$) it is established that the non-ideal Schottky diode-like mechanism limits the current flow in these structures before breakdown, independently of irradiation doses and insulator thickness, while post breakdown J-V characteristics at middle and high voltage ranges is described by power law. If not taking into account of the slight decrease in the effective dielectric constant of the thick stack it may be claimed that E-beam lithography technique can be used to pattern the ultra small MIS devices without degrading the basic electrical parameters of the suggested insulator stacks.

Keywords: Thin Films, Electronic Materials & Processing.

Adsorption pumping revisited

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Abstract

Activated charcoal is broadly used while coupled to a Gifford Mac-Mahon cryocooler's cold finger, performing a cryopump. Such clean high vacuum attracts several applications. Nevertheless, even if it's generally used since the 1960's it does not show up much evolution, discarding the new techniques as miniature cryocoolers and the new porous materials available (metallic foams, new zeolites, Metal-Organic Frameworks, the fashion carbon nanotubes …)

The same charcoal is found in some cryogenic components where a pressure control is critical. It can lead a compression-expansion thermodynamic cycle in a space qualified 300 mK cryocooler [1]. Or it can determine when to have or not good heat conduction between two cryogenic components in a Gas Gap Heat Switch [2, 3], for instance.

Despite of the interest of mentioned components, they are not currently optimized, lacking of characterization of the adsorbants at the desired range of low pressures and temperatures for the working gases.

The work being presented here intends to fulfil this lack, yielding the data to characterize several pairs of gas-adsorbant at sub atmospheric pressure and cryogenic controlled temperature. An experimental setup is described, its validation carried up with helium gas on vegetable activated carbon and compared to published data.

Keywords: Cryopumping, Adsorption

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Construction and test of an electrostatic lens system for ultraviolet photoelectron spectroscopy

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Abstract

In order to improve the resolution and transmission of a gas phase ultraviolet photoelectron spectrometer, an electrostatic lens system, power source apparatus and controlling program were developed [1].

The lens design was based on a simple three element system, with the ability to focus and accelerate/decelerate the incoming photoelectrons, prior to entering a 150º spherical sector electrostatic analyser. Simulation and characterization of both lens and analyzer were carried out using SIMION 3D 7 [2]

The voltages applied to the optical elements were controlled through a specially written LabView program and an electronic circuit developed for this purpose. Ray-tracing studies are performed maintaining constant the linear magnification during the entire range of energies analysed and keeping the angular dispersion of the photoelectron beam to a minimum [3].

Preliminary results showed an effective improvement on the overall working resolution of the spectrometer, which enabled vibrational structure analysis of low ionization energy bands and comparison between band intensities throughout the whole spectrum.

Keywords: Instrumentation; Electrostatic Lens, Ultraviolet Photoelectron Spectroscopy

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Study of depletion of reference leaks: theory versus periodic recalibrations

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Abstract

A study of the depletion of reference leaks, i.e. the loss of the leak rate, is presented. Depletion is one of the most important contributions to the final uncertainty of a reference leak, so its determination is of critical importance to determine final contribution to expanded uncertainty of the leak. The loss of the rate of the reference leak can be determined either by the well-established theory of gas loss of helium in the reservoir through the conductance to the atmosphere or by periodic recalibration at a National Metrological Laboratory usually with annual periodicity. Comparison of both methods for the last eight years are presented and discussed for leaks ranging from 10^{-5} mbar.l/s to 10^{-10} mbar.l/s. Values of depletion from manufacturer are also shown.

Keywords: Vacuum, helium leaks, calibration, depletion

Leak Calibration Method for any Gas at a Variable Pressure

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Abstract

A new method for the calibration of reference leaks at a variable pressure is described. The method is based in a quadrupole mass spectrometer sniffing a closed volume connected to the leak under calibration. Prior to the calibration the volume is pumped and then filled to the desired calibration pressure with N2. Then the concentration of the added gas is continuously monitored by the mass spectrometer. The rate at which the concentration rises is proportional to the leak flow rate.

Calibration of this method may be performed by different ways:

1- With several calibrated leaks of the same gas, keeping constant the experimental conditions;

2- Calibration with one calibrated leak at several known volumes;

3- Calibration with known gas mixtures.

If the third option is used, this method may be used as a primary standard, since calibration is not performed with the same measurand.

Preliminary results have been acquired with helium showing the proportionality between the leak and the concentration rate measured by the quadrupole. With the system used, leaks in the 10^{-5} and 10^{-6} mbar.L/s range where able to be detected. The lower limit depends on the volume, the mass spectrometer sensibility and the sniffer conductance. The upper limit may be easily extended by the use of a larger volume.

The major advantage of this method is its ability to be used with any gas in a wide pressure range. In the case of molecular gases like those used as refrigerants, the only difference in the procedure is that a characteristic mass peak should be selected to measure the concentration. This is the motivation underlying the present work.

Keywords: Vacuum, helium leaks, calibration

On the Permeability of Cork— Preliminary Results

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Abstract

Cork is extensively used for stoppers in wine bottling. Its mechanical properties make it an excellent choice for such use. However, in recent years many wine producers start using other non-natural alternatives. However, it is believed, that cork is not a passive stopper but it helps the evolution of wine.

In the frame of this work, we are evaluating the permeability of cork for gases. The main goal, is to check if its permeability is selective or not, if some gases flow through the cork better than others. Since cork essentially consists of empty cell walls, selectivity may still be present as in living cells.

At this stage, we are presenting only results with helium. Although helium is not a gas related with wine, the permeability may be easily measured with a good helium mass spectrometer leak detector.

Samples were prepared with the shape of small discs of 10 mm in diameter by 2 mm thick. Cuts in different directions were made to evaluate the permeability isotropy.

The first results, showed flows in the range of 10^{-7} to 10^{-4} mbar.L/s. Filling of the empty cells were easily detected. And once these cells were filled with helium, it took many days to pump it out.

The pressure of helium against the cork was varied between 1 and 2 bar absolute. Depending on the pressure, different flows could be identified. At higher pressures, the flow suddenly increased about 1 order of magnitude, suggesting that new flow channels start to open. This process was found to be reversible, showing that such channels were not due to any permanent damage.

More results will be presented and discussed.

Keywords: Cork, permeability

Achievement of pressures from the xhv range by a crypump.

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Abstract

An etalon of pressures from the uhv range that is prepared in Czech Metrological Institute nowadays has need of a pumping unit with the ultimate pressure in the order of 10^{-10} Pa. Since the cryopump was found to be most promising a comprehensive analysis of cryopumping was performed. It results in conclusion that there are two key processes limiting the ultimate pressure of cryopumps: the first one is gas release from cryopump`s surfaces that are not cooled to the cryogenics temperatures and the second one is the temperature of the cryopanel the gas is condensed or physisorbed on. To reach the pressure from the xhv range the walls of the cryopump that are not cooled to the cryogenics temperatures (the “envelope”) have to be outgassed at an elevated temperature and the temperature of the cryopanels should be as low as a few Kelvins. Moreover, the partial pressures of helium and neon have to be lowered sufficiently during preliminary pumping. For that reason, pumping from atmospheric pressure has to be accomplished by a transport pump to an adequately low pressure.

On the basis of these consideration a cryopump was designed which could be baked together with the whole vacuum system at a temperature higher than 250°C and is equipped with a refrigerator which is capable to keep the temperature of the cryopanel at a temperature 5 K approximately. As a forepump is utilized a turbomolecular pump that allowed achieving a pressure in order 10^{-8} Pa in the pumped vacuum system before the cryopump is activated.

Besides of the complete pump design some details of pump construction will be presented as well.

Keywords: cryopump, xhv

Nanocrystalline diamond films grown by HFCVD in rgon-rich atmospheres

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Abstract

Nanocrystalline diamond (NCD) has been object of several studies due to its intrinsic properties, namely high hardness, high wear and corrosion resistance, low surface roughness, biotolerance and high self-friction coefficient, which enable a great variety of applications: optical, mechanical, electronic and biomedical. Synthesis of NCD is usually carried out by chemical vapour deposition (CVD) techniques using a reactant gaseous mixture that includes a carbon source and hydrogen (H_2). One approach to grow NCD instead of microcrystalline diamond (MCD) by CVD is the partial or complete replacement of H_2 by argon (Ar). Among the CVD techniques, microwave plasma (MWCVD) is the most used method to deposit NCD using argon-rich atmospheres. However, due to the low capital cost and the ability to scale up, hot filament chemical vapour deposition (HFCVD) system has advantages over MWCVD in producing low cost diamond films. Furthermore, HFCVD is able to coat complex shapes and internal surfaces, which is useful for specific applications.

In the present work, the deposition of NCD films by HFCVD using large percentages of Ar, is reported. Different surface pretreatments used to improve diamond nucleation are studied in order to achieve homogeneous and dense NCD coatings at moderate growth rates. Conventional diamond seeding by ultrasonic bath of diamond powder (1g) and n-hexane (100 ml), Rotter pre-treatment [1] and manual scratching were the surface treatments that were carried out on silicon (Si) substrates. NCD deposition conditions were the following: filament temperature = 2100-2300ºC, substrate temperature = 650 – 750ºC, total gas pressure = 25-100mbar, total gas flow = 25-200 mlmin-1, Ar/H_2 = 1-6, CH_4/H_2 = 0.03-0.07. Diamond films were characterized by Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), X-ray diffraction (XRD) and Raman-UV spectroscopy.

Keywords: nanocrystalline diamond; HFCVD; argon-rich atmosphere

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Ti_xSi_yN Nanocomposites by Cathodic Arc Plasma Deposition

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Abstract

Cathodic arc deposition is a technique that allows synthesizing hard thin films that can be used as protecting coatings for a number of applications. Owing to the great interest posed by this kind of films, we believe that it is important to determine not only the deposition conditions necessary to obtain nanocomposites with good mechanical and tribological properties, but also the way in which the mechanisms of the process influence those properties.

We have synthesized ternary TixSiyN nanocomposites by means of a custom-made cathodic arc system in which a TiSi 80/20 at% target has been installed, using a mixture of argon and nitrogen. Different deposition parameters (such as the total pressure in the vacuum chamber, the deposition temperature, the amount of nitrogen in the gas mixture, or the arc current) have been changed in order to study their influence on the properties of the obtained thin films. These have been characterized by means of techniques like profilometry, X-ray diffraction (XRD), glow discharge optical emission spectrometry (GD-OES), scanning electron microscopy (SEM), nanohardness measurements or pin-on-disc testing.

XRD analysis shows that the TiN nanocrystals in the films present a preferred (200) orientation in all cases. The degree of texture, defined as the ratio of the (200) peak to the (111) peak intensities, depends mainly on the amount of nitrogen in the gas. The content of silicon in the films, determined by GD-OES, is below 5.5% in all the films under study. The surface roughness of the films, measured by means of a profilometer, does not depend on the temperature applied to the substrate before the process.

Keywords: ternary nanocomposites, cathodic arc deposition, hard coatings

Influence of the aluminum incorporation on the structure of sputtered ZrN_x films deposited at low temperatures

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Abstract

Zirconium nitride has attracted much attention because their suitable structural, optical and mechanical properties for applications on cutting tools, in surgical implants, as protective coatings and in decorative purposes due to it has high hardness (up to 23.5 GPa in the best case), gold color and high corrosion resistance, among others. The aluminum is an element that enhances the toughness and hardness when it is incorporate into a nitride as a impurity. For these reasons, we have studied the influence of the Al incorporation in the crystalline structure of ZrN thin films deposited by dc magnetron sputtering processes at low temperature. The incorporation of the aluminum in the films depends directly from the Ar/N_2 ratio in the gas mixture and the power applied in the aluminum cathode during the deposition. Chemical composition and crystalline structure of the films were analyzed by Energy Disperssive X-ray spectroscopy (EDX) and X-ray Diffraction (XRD), respectively. When Al atoms are incorporated in the ZrN coatings, the strong ZrN (200) preferred orientation is modified to a combination of phases related to (111) ZrN with a contribution of hexagonal (100) AlN, cubic (111) AlN and possibly (211) Zr_3N_4 which are detected by XRD for high aluminum concentrations. FTIR spectra (Fourier Transform Infrared) allowed us to complete the identification of the nitrides and oxides incorporated in the deposited films. The effect of a bias voltage applied to the substrate has also been investigated and related to the changes in the microstructure and nanohardness measurements of the ZrAlN films.

Keywords: ZrAlN, ternary coatings, magnetron sputtering, hard coatings

Effect of the Oxygen Contamination on the Structural and Mechanical Properties of AlC, AlN, and AlCN Thin Films Obtained by R.F. Magnetron Sputtering

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Abstract

Aluminum carbide (Al-C), aluminium nitride (Al-N), and aluminum carbonitride (Al-C-N) thin films were grown onto Si [100] substrates by r.f. reactive magnetron sputtering at 400°C with intentional high base pressure to introduce impurity oxygen from residual air in the chamber. The Al-N coatings were obtained by using an Al (99.9 %) target in Ar/N_2 atmosphere and the Al-C and Al-C-N by using a binary (50% Al, 50%C) target in argon and in Ar/N_2 mixture, respectively. The d.c. bias voltage was varied between 0 and -150 V. The films were characterized by X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDS), Fourier transformed infrared spectroscopy (FTIR) and the mechanical properties by nanoindentation. The structure of the films has been determined by XRD, which shows that amorphous films are formed in all cases. The variation of polarization bias voltage produced chemical differences in the films. As the bias voltage is increased, the Al content is reduced in all three materials. The nitrogen content also varied between 3 and 4%at. for Al-N coatings, remaining practically constant (8%at.) for the Al-C-N films. The Berkovich hardness results were 7.0, 17.2 and 9.2 GPa for Al-C, Al-N, and Al-C-N films, respectively.

Keywords: Aluminum carbide; aluminum nitride; aluminum carbonitride; magnetron sputtering.

Development of novel hard metallic coatings on 316 L steel by hot dipping in Al-Si alloy

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Abstract

In this work we investigate the coating of the 316 LVM steel by hot dipping in an Al-12 Si alloy melted at about 760ºC and using various immersion periods of time. Following immersion, the steel become coated with a tightly adherent, relative thick (~ 15 µm) and even metallic coating, the thickness of which slightly increases with increasing immersion time. The forming coating has been identified as an intermetallic phase containing (at%) 70 Al, 11Fe, 11 Si, 5 Cr. Hardness of the coating of about 850 HV was measured for the earlier stages of immersion. With progressing immersion and after 900 s, hardness of the coating slightly decreases till about 600 HV. Both values are much higher than those obtained in the bulk of the stainless steel (~200 HV), which nearly coincides with the hardness value for the standard 316 LVM steel. To evaluate the corrosion resistance a set of electrochemical tests were conducted by using a physiological solution that contains a relative large concentration of Cl- ions, which make the medium very aggressive. Impedance diagrams were obtained just up to 1 week of immersion. Preliminary results suggest that the coated steel is able to self passivate, exhibiting a corrosion behaviour that is similar to that of the non coated steel. The biocompatibility of 316 LVM steel, before and after hot dipping in Al-12 Si alloy, was analysed by using human cells related to the osteoblastic phenotype. Preliminary results indicated that cells exhibit a well-organized actin cytoskeleton on both surfaces. Cell spreading was unaffected by metallic coating on 316 LVM steel samples. In summary, improved mechanical and corrosion behaviour together with a good in vitro biocompatibility make this novel hard metallic coating a promising modification of 316 LVM steel for biomedical applications.

Keywords: Stainless steel 316 LVM, Hot-Dipping, Coatings, Biomaterials

Deposition of diamond films using a high argon concentration in HFCVD system

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Abstract

Diamond particles with different grain sizes were deposited using a hot filament chemical vapor deposition (HFCVD) method at very high argon concentrations (75 and 90 vol%) into mixtures of methane and hydrogen. The pressure was regulated at 30 Torr and the substrate temperature was held constant at a set point value ranging from 550ºC and 800ºC by external heating. High resolution scanning electron microscopy (FEG-SEM), Raman spectroscopy and high resolution x-ray difractometry were used to characterize the surface morphology, carbon phases and crystalline properties. The results reveal a dramatic change in the surface morphology of films grown at different argon concentration. Micro (MCD) and nanocrystalline (NCD) diamond films were obtained using 75 vol% of argon and ultrananocrystalline (UNCD) diamond films were obtained using 90 vol% of argon. The activation energy, determined from the Arrhenius plot, was measured using mass growth rate. The value obtained for 75 vol% of argon, around 10 kcal/mol, is the same obtained for MCD in previous work [1]. This value can be an evidence that the growth mechanism of NCD in HFCVD reactors is very similar to MCD growth, as proposed by other authors [2-3]. The value obtained for 90 vol% of argon, around 6 kcal/mol, is the same obtained for other authors [4-6] being an evidence that the growth mechanism of UNCD is different from that of MCD and NCD. In this work we compare and contrast the growth and properties of the films obtained.

Keywords: nanocrystalline diamond, ultrananocrystalline diamond, HFCVD, argon, activation energy

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Correlation between carbon content and mechanical properties in ZrCN coatings for tribological applications

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Abstract

In recent years many efforts have been made with the aim of increase the performance of many mechanical components with regard to the tribological applications. The most important drive force is the continuous growing industrial demand for enhanced performance of many mechanical components in terms of friction and wear. In this work we aim to contribute in such area, particularly by studying properties of ZrCN coatings (deposited through the technique of dc reactive magnetron sputtering). In order to investigate the influence of the amount of carbon incorporated in the coating, films were grown at different C_2H_2 flows, ranging from 2 to 10 sccm. The Ar and N_2 flows were kept constant at 60 and 8 sccm, respectively. Glow discharge optical emission spectroscopy was used for the determination of the chemical composition of the coatings.

The carbon content in the coatings increased from 8 to 26 at. % while a decrease of the Zr content from 19 to 12 at.%. On the other hand, with the increasing of C_2H_2 flow was observed a decrease of the N content from 68 to 61 at.%. Structural investigations by means of X-ray diffraction suggest the formation of a mixture of fcc phases of ZrN, ZrCN and ZrC, (NaCl-type). Hardness and Young's modulus as measured by nano-indentation ranged from 11-22 GPa and 225-244 GPa, respectively. Since main focus was laid on the tribological properties, ball-on-disk tests were performed at room temperature. Here, the coefficient of friction (from 0.4 to 0.1) as well as the wear rate (from 1.1×10^{-15} to 1.3×10^{-16} $m^3N^{-1}lap^{-1}$) decreased with increasing C_2H_2 flow during deposition. In summary, the presented work elucidates the relation between microstructure, mechanical and tribological properties of ZrCN coatings, deposited by dc reactive magnetron sputtering.

Keywords: Multifunctional coatings, tribological behaviour

Ab Initio Study of (Ti,Si,Al)N Metastable Phase

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Abstract

(Ti,Si,Al)N coating materials have recently been developed as an alternative to traditional coatings, due to its unique properties. Thin films within the (Ti,Si,Al)N system were prepared by dc and rf reactive magnetron sputtering, showing a mixture of crystalline phases with fcc type structure. One of them corresponds to a Ti–Si–Al–N phase where some of the Si and Al atoms are occupying Ti positions in the TiN lattice.

The aim of the present work is to study the electronic, structural, and mechanical properties of (Ti,Si,Al)N metastable phase, using first principles calculations based on the density functional theory. These calculations provide the lattice parameter, density of states, cohesive and formation energy, elastic constants and moduli, when Si and Al atoms substitute Ti in the TiN lattice. The calculated values are generally in good agreement with experiments and compare well with other theoretical results.

Keywords: Titanium, Hard Coating, Density Functional Theory

Nano and Micro Indentation and Scratch Tests of Mechanical Properties of Hard Coating

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Abstract

Experimental comparison of common nano and micro scales for hardness evaluation has been performed on coating samples and discussed in details. Instrumented indentation was found advantageous in both repeatability and a number of measured parameters over classical hardness methods for metals, while sclerometry was advantageous for ultra-thin films.

Introduction

Traditional macro-hardness tests of bulk materials are conducted at high loads of the order of kilo-Newtons with post-test indent analysis. Micro-hardness measurements of relatively-thick coatings and bulk materials are performed under the medium loads of the order of deca-to-hecto-Newtons. As the industrial technology advanced, the mechanical characterization went to nano levels, with the loads used for nano-indentation tests of thin films down to micro-to-milli-Newtons. A big step forward has been in-situ continuous force-depth monitoring during loading and unloading parts of an indentation cycle [1], which allowed for both instrumented-hardness and Young's modulus evaluation using equations like that of Oliver-Pharr.

In all the indentation tests, pressure distribution is concentrated under (in the front of) the indenter and thus makes it vulnerable to substrate effects for ultra-thin films. Thus, advantageous for thin films may be scratch-hardness tests with the pressure distribution (still in the front of the indenter) in the same surface layer where the indenter is sliding. As post-scratch detection of the shallow nano-scratches is challenging, utilization of the same tip for both scratching and nano-imaging may be the best way for nano-scratch-hardness testing of ultra-thin films.

This paper compares different techniques for micro and nano indentation and scratch tests on hard coating samples, touches on the fundamental constraints of the current techniques, and throws light on new technologies. It has been done mostly on hard coating, but its techniques and conclusions are applicable to various materials.

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Deposition of TiN/CrN multilayers with nanometric bi-layer periods by Cathodic Arc PVD

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Abstract

The look for ultrahard and high wear resistant coatings has led to PVD coatings with multilayer structures in the nanometric range. In this line, PVD coatings alternating TiN and CrN layers and different bi-layer periods have been deposited on high speed steel and Silicon substrates, for investigating the mechanical and tribological properties and study their correlation with the bi-layer period. The coatings were deposited by cathodic arc in a semi-industrial PVD equipment and their total thickness was about 1.5 microns, and their mechanical and tribological properties were studied. The coatings were analysed by GD-OES, achieving quantitative in-depth profiles that could only reveal the multi-layered structure for the highest periods. A cold cathode FE-SEM equipment was also used for the observation of the structures. The images obtained revealed the nano-layered structures in all the cases, showing bi-layer periods in the nanometre range. The nanoindentation tests showed that coatings with lower bi-layer periods tended to present higher hardness values.

Keywords: PVD, Multilayers, Titanium, Chromium, Hardness, Wear, Friction, GDOES, FE-SEM

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In-situ, Real-time Observation of Thin Film Deposition: Roughening, Zeno, Grain Boundary Crossing Barrier, and Steering

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Abstract

Thin polycrystalline metal films are becoming increasingly important, as is reflected in the multitude of applications in nanotechnology, nanooptics, microelectronics, vacuum coating, catalysis, medical science, sensor elements, wear protection layers, decorative coatings, and the synthesis of new materials.

As thin film properties are intrinsically linked to the precise film structure, one would like to control the overall film morphology down to the nanometer scale. This clearly demands fundamental research that links well-known atomic processes, such as diffusion and nucleation, with the mesoscopic film evolution during film growth.

Applying video-rate Scanning Tunneling Microscopy (STM) [1], we succeeded in visualizing film growth with atomic-scale resolution in real-time. We evaporated several tens of monolayers of gold on top of a well-annealed polycrystalline gold film, while continuously observing the evolving surface with the microscope. These measurements directly visualize atomic processes that take place during film growth.

Analyzing the evolving film structure, we observe a significant increase in the film roughness, which we explain by considering both “well-known”, single crystalline growth modes in combination with additional polycrystalline effects [2]. The grain boundaries play a crucial role in the evolution, as they initiate mound formation, thereby significantly increasing the total film roughness. A possible additional roughness contribution comes from atom steering, which also can delay the film closure in the early stages during film growth.

We expect that our findings are of a very general nature and that the processes described in this talk will occur on almost every polycrystalline film during both its nucleation and growth.

Keywords: Film growth Polycrystalline (thin) films Growth modes and evolution In-situ, video-rate STM

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In-situ diagnostics in VHF PECVD process: A crucial aspect for fast fabrication of thin film silicon solat cells

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Abstract

For thin film silicon solar cells to compete with c-Si and other types of thin film materials such as CdTe and CIS, the deposition rate has to be increased substantially compared to the present status at the industries. Several deposition techniques promise to deliver that, however, the thin film Si, especially nanocrystalline Si, made by VHF PECVD has so far shown the best results, in terms of efficiency as well as deposition rate. This can be attributed to the available ion energy that can be tuned by pressure, coupled power, process temperature, plasma frequency [1] and external applied voltage bias to the RF electrode. We present here how to find a suitable combination of these parameters to attain high growth rates (>5nm/s). The second optimization step is the identification of the so called crystalline to amorphous transition regime which in a plasma process can be achieved by varying delivered power and/or hydrogen dilution. The third optimization step is the gas distribution in the plasma zone, which is achieved by a suitable showerhead design. The fourth optimization step involves a deposition process that avoids the progress toward the dusty plasma regime. Use of smaller inter-electrode distance and Amplitude modulation of the RF plasma are proposed as candidates to achieve this. This step also has a consequence on homogeneity of the film. We will show how various diagnostic tools, such as optical emission spectroscopy, V-I plasma probe and ion energy analyzer can be used in the optimization process to obtain state of the art nc-Si solar cells with high deposition rates.

Keywords: Nanocrystalline silicon, PECVD, Thin films, Solar cells

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Structural and optical properties of nitrogen doped ZnO films

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Abstract

Zinc oxide is getting an enormous attention due to its potential applications in fields such as optoelectronics, spintronics and sensors. The renewed interest in this wide band gap oxide semiconductor relies on its direct high energy gap (E_g~3.437 eV at low temperatures) and large exciton binding energy ZnO. These characteristics make ZnO a very promising candidate for optoelectronic based light emitters in the blue and ultraviolet spectral range in competition with GaN.

To reach the stage of device production some important issues must be solved. The doping process must be reproducible, particularly p-type doping, and the growth quality (defects and contaminations). Among the potential p-type dopants nitrogen is being considered as one of the most important candidates.

In this study we discuss the structural and optical properties of nitrogen doped ZnO during the growth. The films were deposited by magnetron sputtering using different conditions and substrates. Some selected samples were post annealed to assess the stability and recrystallization kinetics. The composition and structural properties of the as-grown and annealed films was studied combining grazing x-ray diffraction (XRD), Rutherford backscattering (RBS), heavy ion elastic recoil detection analysis (HI-ERDA).

The results clearly show an improvement of the films quality with the deposition temperature and a stable homogeneous composition through the entire probed thickness (~2 μm). The ERDA analysis reveals the presence of H on the samples ZnO. Annealing in a reducing atmosphere increases the concentration of H in the films. The crystalline quality of the films improves with the deposition temperature. These is observed by the narrowing of the diffraction peaks and confirmed by the Raman modes. The photoluminescence also evidence the band edge emission of ZnO and the red band associated with residual defects. Hall measurements reveal transient p-type behaviour in some of the films.

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Pulsed laser deposition of oxide based thin films for spintronics

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Abstract

Pulsed Laser Deposition (PLD) is a widely used technique for the growth of thin films due to its versatility, reliability and ability to control the stoichiometry of complex multicomponent materials. Furthermore, it is often possible to grow thin films with improved physical properties at temperatures as low as room temperature.

In this work, we will discuss the preparation of thin films of oxides by PLD and their structural and physical properties as a function of processing parameters. In particular, we will concentrate on materials with interest for applications in spintronics such as half-metallic ferromagnetic oxides and dilute magnetic oxide semiconductors.

Keywords: PLD, dilute magnetic semiconductors

New trends in decorative and functional coatings

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Abstract

Industrial manufacturers of ceramic tiles, window glasses, car and house accessories or domestic appliances are increasingly interested in new decorative coatings, deposited by cleaner processes. Beyond the prevention of pollution, there is an opportunity to reach a multiple goal: new colours and optical effects, higher resistance to wear or corrosion and, even more important, the possibility of providing new functionalities to the coated surfaces like hydrophobic, self-cleaning, biocide or hypoallergenic properties.

Vacuum coating technologies, like PVD are the main candidate to provide reliable and versatile methods to coat a broad range of material substrates, including metals, ceramics, glasses or polymers. In order to achieve mass-productive industrial processes there are some challenges to be dealt with, as the implementation of homogeneous large area or continuous processes or the temperature control in the case of low melting point substrates.

To meet this goal, a new research project in the decorative-functional coating field is running from the beginning of 2007. This project, titled CENIT ART-DECO (Advances in Decorative-and Functional Coatings), 14 Spanish companies and 12 research institutes will collaborate during four years (2007-2010), investing a budget of 25 million Euros.

This paper details the research lines and the early results of the ART-DECO project. Objectives, methodology and the preliminary advances in the different strategies are summarised.

Keywords: Decorative Coatings, Functional Coatings

Oxidation behaviour of CrAlN, AlTiN and nonocomposite coatings

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Abstract

CrAlN, AlTiN and Chromiun base nanocomposite coatings were subjected to thermal annealing cycles in an atmosferic furnace. The tribomechanical properties, compositional and chemical changes were analysed in order to study their oxidation behaviour and thermal stability.

CrAlN coatings with different Cr-Al stoichiometry were deposited by using of a cathodic arc PVD machine. The behaviour of these coatings has been compared with the oxidation performance of nc-CrAlN/Si_3N_4 nanocomposite coating and with the more conventional TiAlN coating deposited in rotary arc PVD equipment.

Ultramicrohardness, friction and wear resistance measures were carried out before and after thermal annealing in order to study the evolution of the tribomechanical properties. The oxidation process, at different temperatures was studied by means of GDOES analysis looking for the evolution of the composition in depth for the oxygen and other elements and the chemical state were analysed by means of XPS. These profiles and the microstructure of the coatings were also investigated by FE-SEM.

As a conclusion this paper looks for the relations between the microstructure, compostion and tribological properties of the different coatings before and after the oxidation experiments.

Keywords: Oxidation, CrAlN coatings, tribology. nanocomposite

Effect of O Addition on the Thermal Behaviour of Hard W-N Sputtered Coatings

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Abstract

Among transition metal nitrides, coatings of W-N system have been recently studied due to the outstanding high temperatures mechanical properties of W-based materials. However, it is also well known that W-compounds have low oxidation resistance, particularly for temperatures higher than 600ºC. One of the possibilities to overcome this drawback would be the addition of W-N coatings with oxygen, with the final scope of favouring the formation of a protector W-O layer. However, O contents have to be low in order not to degrade the intrinsic mechanical properties of the nitride phases.

Thus, coatings from W-O-N system were deposited by dc reactive magnetron sputtering from a pure tungsten metallic target, onto Ferroalloy steel substrates, using an O_2-N_2 gas mixture and their properties compared with pure tungsten nitride coating. The total coatings thickness was kept approximately constant, $\approx$ 2.5 μm, and the oxygen content, evaluated by EPMA, varied from 24 to 30 at.%. The structural changes, as a result of the coatings heating up to 900ºC, were characterized in-situ by High Temperature X-Ray Diffraction (HT-XRD). Furthermore, ex-situ annealing in protective environment at increasing temperatures up to 800ºC was performed and the coatings were characterised in relation to their structure, chemical composition and hardness. Generally it could be said that the minor O content the best is the W-O-N thermal behaviour. In addition to structural and compositional stability the $W_{32}O_{24}N_{44}$ film presents a hardness increase of ~ 45% after thermal treatment at 700ºC in comparison to the as-deposited state. However, an inverse trend was detected after post-annealing at 800ºC, which was more evident for the high O content films. The sharp decrease in hardness came with compositional variation due to N_2 effusion from the coatings.

Keywords: Tungsten oxynitride; sputtering; thermal behaviour; HTXRD; hardness

The Nano Modification of Duplex Hard Coatings with Nitrogen Ion Implantation

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Abstract

In this work, the mechanical properties of new hard coatings based on a multilayer structure have been investigated at the nanometre scale. A multilayer structure of nitrided layer/TiN/ion iplalantation on steel substrate has been deposited by Physical Vapor Deposition and Ion Beam Assisted Deposition. The nanomechanical properties have been determined measuring nanoindentation, friction and wear by means of an atomic force microscope (AFM) on the multilayer.The microstructure of obtained nitrided layer showed differences with regard to the presence or absence of a white layer, its thickness and its ε/γ′ phase ratio (XRD) and nitriding depth. In the present investigation the subsequent ion implantation was provided with N^{2+} ions. This paper describes the successful use of the nanoindentation technique for determination of hardness and elastic modulus.The results are analyzed in terms of load-displacement curves, hardness, Young's modulus, unloading stiffness and elastic recovery. The analysis of the indents was performed by Atomic Force Microscope. The analyzed AE signal was obtained by a scratching test designed for adherence evaluation. Coating is often in tensile stress with greater microhardness. The stress determination follows the conventional $\sin^2\psi$ method, using a X-ray diffractometer. The (422) diffraction peak was recorded in a 2θ interval between 118^o and 130^o, with tilting angle. The evolution of the microstructure from porous and columnar grains to densel packed grains is accompanied by changes in mechanical and physical properties. Therefore, by properly selecting the processing parameters, well-adherent films with high hardness can be obtained on engineering steel substrates. All these results indicate that the multilayer combines ultrahard behaviour with good tribological and wear properties.The experimental results indicated that the mechanical hardness is elevated by penetration of nitrogen, whereas the Young’s modulus is significantly elevated.

Keywords: films, nano, PVD, IBAD, Ion implatation

Elastodynamics of Inorganic and Polymeric Sculptured Thin Films

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Abstract

Sculptured thin films (STFs) are nano-engineered, porous, solid thin films conceptualized in the early 1990s and fabricated by several research groups since then [1]. The nanostructure of inorganic STFs typically comprises 1-3 nm dia clusters arranged to form parallel nanowires that are bent in some fanciful forms with feature size 30 nm or larger. Although most STFs are made of inorganic materials, some polymeric STFs have also been fabricated in recent years.

The Mori-Tanaka average stress and the Eshelby tensor for ellipsoids has been used within the framework of localized homogenization in order to estimate the elastic constitutive properties of an STFs from its morphology. The devised model contains five arbitrary parameters, whose values can be decided by suitable experimentation. Composite compliances governing the elastostatic and the elastodynamic responses of chiral STFs to axial excitation depend on certain morphological features, as also are the characteristics of the polarization-discriminatory Bragg phenomenon associated with chiral STFs.

The optical properties of polymeric STFs can be controllable piezoelectrically. This is exemplified by the shift of the Bragg center-wavelength of a polymeric chiral STF by the axial tension generated in a co-bonded piezoelectric disk by a dc voltage. This attractive possibility can be exploited for tunable optical filters as well as lasers made of chiral STFs, and can be extended to other types of STFs.

Keywords: Bragg phenomenon, local homogenization, piezoelectric control, sculptured thin films, structural chirality, tunable optics.

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